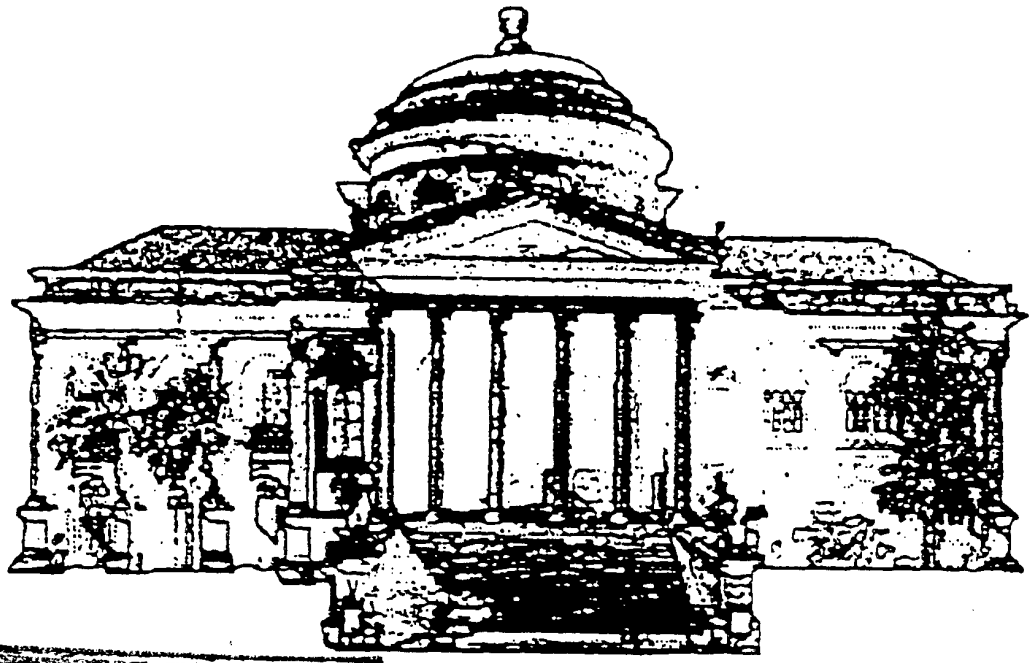
3rd EUROPEAN SYMPOSIUM on X-RAY TOPOGRAPHY and HIGH RESOLUTION DIFFRACTION

X-TOP '96



PALERMO, ITALY

Hotel Torre Normanna

22-24 April 1996

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ABSTRACTS OF ORAL CONTRIBUTIONS

RECIPROCAL SPACE MAPPING FOR SEMICONDUCTOR DEVICE HETEROSTRUCTURES

Mark S. Goorsky
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We use reciprocal space mapping, double axis diffraction, and x-ray topography to assess the relationship between the crystalline structure of semiconductor heterostructures and the performance of devices fabricated from these structures. Results from two device structures will be presented.

Pseudomorphic high electron mobility transistors (pHEMTs) require the synthesis of strained conductive channel regions which are free from misfit dislocations. Using advanced x-ray techniques with electron microscopy, we demonstrate that the substrate perfection in GaAs-based devices plays a strong role in the formation of misfit dislocations; the dislocations, in turn, impair device performance only when an orthogonal array of misfits form. Substrates with higher initial levels of perfection -as determined through whole wafer reciprocal space mapping - allow for the growth of more highly metastable layers. However, extrinsic defects such as particulates introduce heterogeneous misfit dislocation nucleation sites as determined with x-ray topography. The recent drive towards the use of InP-based pHEMT stems from the higher device performance anticipated from channel layers with higher conductivity than is possible for GaAs-based structures. Our recent results demonstrate that whole wafer reciprocal mapping represents a useful means to assess the quality of InP wafers prior to epitaxial layer deposition and we have found that the lower stability of InP-based structures (compared to GaAs-based structures) is directly attributable to the lower overall crystalline quality of the InP substrates.

The non-destructive nature of the x-ray techniques and their high sensitivity to defects which lead to device degradation makes these techniques ideal for studying structure/performance relationships in semiconductor heterostructures.

X-RAY REFLECTOMETRY RECIPROCAL SPACE MAPPING OF STRAINED SiGe/Si SUPERLATTICES

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The perfection of the interfaces in strained SiGe/Si superlattices is an essential parameter for their optical and electrical properties. It has been reported previously [1] that the interface roughness of these structures exhibits both in-plane and inter-plane correlations. The in-plane correlation results in a terrace structure depending on the miscut angle of the substrate, and due to the inter-plane correlations, the sequence of the terraces is nearly perfectly replicated during the superlattice growth.

X-ray reflectivity measurements enable the study of both types of roughness correlation. From the distribution of the non-specularly reflected intensity in reciprocal space the in-plane correlation function of the roughness profile as well as the degree of the inter-plane replication can be derived. The in-plane correlation functions of various types of terraced interfaces are derived theoretically using the approach of stationary random processes [2]. On the basis of these correlation functions and a phenomenological description of the inter-plane replication of the terraces [3] we simulated the reciprocal space distribution of the non-specularly scattered intensity using the distorted-wave Born approximation.

In the experimental part of the paper, we report on the synchrotron measurements of the non-specular x-ray reflectivity of SiGe/Si superlattices grown with MBE under various growth conditions. The measured reciprocal space distribution of the non-specularly scattered intensity has been compared with the theory and a qualitatively good agreement has been achieved. We have found distinct maxima of the non-specularly scattered intensity in the reciprocal plane. The average terrace dimensions have been obtained from the positions of these maxima. It has been demonstrated that the orientation of the terraces and the terrace widths are correlated with the miscut angle of (001) planes in the substrate. The terrace shape as well as the extent of its replications could be deduced from the analysis of the shape of these maxima. This analysis made it possible to choose the most suitable structural model of the interfaces.

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[3] E. Spiller, D. Stearns, and M. Krumrey, J. Appl. Phys. **74**, 107 (1993).

Lateral periodicity in highly-strained (GaIn)As/Ga(PAs) superlattices investigated by x-ray scattering techniques

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Symmetrically strained (GaIn)As/Ga(PAs) superlattices grown on off-oriented InP substrates are challenging new materials in the research field of highly-strained III/V semiconductor heterostructures. This material system offers the possibility of tailoring changes in surface morphology as a function of the strain incorporated in the epilayer. This is an extremely interesting possibility since, recently, a close relation between surface morphology and strain relaxation has been pointed out.

In this work we investigate the lateral periodicity of several symmetrically strained (GaIn)As/Ga(PAs) superlattices by means of x-ray scattering techniques. The multilayers were grown by metalorganic vapour phase epitaxy on InP substrates intentionally off-oriented towards one of the nearest {110} directions (miscut angle=0-4 deg). Several high-resolution triple-crystal reciprocal space maps are reported for different azimuth angles in the vicinity of the (004) Bragg diffraction and countor maps of the specular reflected beam collected in the vicinity of the (000) reciprocal lattice point are shown. The reciprocal space maps clearly show a two-dimensional periodicity of the x-ray peak intensity distribution which can be ascribed to the superlattice periodicity in the direction of the surface normal and to a lateral periodicity in a crystallographic direction related to the miscut angle. Simulations of the two-dimensional profiles allow us to determine the thicknesses and the strain modulation of the superlattices both in the growth direction and in the interface plane. The lateral periodicity is found to increase with the decreasing of the miscut angle. An onset angle for the appearance of the lateral modulation is given. At the highest miscut angles a curvation of the lateral strings is detected and explained by surface roughening. The results of low-angle countor maps are compared and discussed together with atomic force microscopy investigations.

X-ray diffraction from layered surface gratings: investigation of grating induced strain

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Epitaxial single layer and multilayered surface gratings have been investigated by high resolution X-ray diffraction, mapping the fine structure of the diffraction intensity in reciprocal space in the vicinity of different RLP. The diffraction intensity is concentrated along grating truncation rods equidistant and perpendicular to the sample surface. The intensity pattern along the truncation rods represent the grating shape function, the compositional profile and the strain field of the grating. The presence of the surface grating causes the generation of laterally periodic strain. We have studied the influence of the lattice strain field generated by the surface grating on the diffraction pattern. The strain field represents the grating periodicity, depends on the shape of the surface grating and on the lattice mismatch. Within the paper we will give a short theoretical description of the diffraction process and experimental examples for various cases (simple grating, strained layer grating, multilayered grating).

X-RAY REFLECTIVITY AND HIGH RESOLUTION DIFFRACTION FROM III-V SURFACE GRATINGS

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Technological patterning of periodic gratings at the surface of III-V semiconductor layers, including lithography with subsequent etching, followed by a burying growth step, are nowadays frequently required in the engineering of optoelectronic devices. These fabrication methods can induce structural or morphological defects in the corrugated surfaces that are detrimental for the performances of the devices. Recent experiments [1-3] on corrugated crystalline substrates have shown that X-ray diffraction is a very promising technique for the investigation of these types of laterally periodic arrays. Moreover in the case of buried structures this technique could be advantageous with respect to scanning-electronic microscopy, because it is non destructive and gives informations over a large area of the sample.

By means of a combination of X-ray reflectivity and triple axis diffraction experiments we have investigated InGaAsP corrugated layers on InP substrate, before and after burying in InP. We are able to extract the lateral period, the etching depth, the dimensions characterizing the shape of the grating, and surface roughnesses. On the basis of the X-ray data we will discuss the modifications of the interfaces after burying. We will also compare the reflectivity spectra obtained from different azimuthal orientations and discuss the informations we can deduce on the characteristics of the gratings.

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HIGH-RESOLUTION X-RAY DIFFRACTION FROM STRAINED IMPERFECT HETEROSTRUCTURES

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Strained semiconductor thin films are the basis of modern microelectronics and optoelectronics. Strain-induced modifications of electron band structure strongly influence electronic and optical properties of semiconductor devices. The strain state of a crystal lattice is deduced from the measured X-ray diffraction rocking curves by subsequent fittings using dynamical diffraction theory. Simulations based on Takagi-Taupin equations [1] are most popular in this field and provide excellent results in the case of nearly perfect structures, e.g. such as SiGe/Si heterostructures grown by molecular beam epitaxy. However, this powerful method meets difficulties when applied to imperfect structures having a large number of point and extended defects and possessing diffused concentration profiles. As typical examples, the CdHgTe/CdTe heterostructures grown by metal-organic vapor phase epitaxy or the SiGe/Si structures grown by ion assisted beam sputtering, may be mentioned. In this case the number of fitting parameters increases and it is difficult to avoid ambiguity of the fitting procedure. Most important, the Takagi-Taupin equations are not well suited to the description of X-ray diffraction spectra, if short-range fluctuations of lattice parameters have be taken into account.

In this paper a kinematic approach to rocking curve calculations is developed, which is based on a direct summation of waves scattered by atomic planes of the multilayered structure. Kinematic calculations permit the introduction of lattice imperfections into each atomic plane. A problem of an infinitely thick substrate is solved using an exponentially decaying amplitude of the incident beam, due to both extinction and absorption effects [2]. Analytical expressions of the rocking curve shapes were obtained for heterostructures having local fluctuations of the interplane spacings as well as the lateral fluctuations of the thin film thickness, imitating the interface roughness.

The approach developed is used to simulate high-resolution X-ray diffraction rocking curves which were taken from SiGe/Si heterostructures grown by ion assisted beam sputtering. The depth-resolved strain profiles derived as a result of the fitting procedure differ considerably from those which may be concluded using Takagi-Taupin simulations. The obtained results are discussed in the framework of the dynamics of point and extended defects arising during the growth of the samples.

[1] P. Fewster, Semicond. Sci. Technol. 8 (1993) 1915.
[2] E. Zolotoyabko, Y.Finkelstein, M.Blumina and D.Fekete, Physica B, 1995, to be published.

High Resolution X-ray Scattering From CdMnTe/CdTe Multiple Quantum Well Structures

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Optical measurements and theoretical studies indicate that the effects of surface roughness and interfacial state of CdMnTe/CdTe quantum well structures are playing a key role in the optical and transport properties of related devices. Hence, a characterisation of the surface and interfacial structure by means of X-ray techniques is valuable.

A 15 period $Cd_{0.93}Mn_{0.07}Te$(15nm)/CdTe(5nm) multiple quantum well structure grown by MBE on a InSb (001) substrate covered with a 100 nm CdTe buffer layer was investigated by X-ray grazing angle reflectivity, scattering and triple axis diffraction. X-ray scattering intensities near the (000) and (004) reciprocal points, as well as the specular and off specular reflectivities were measured at station 2.3 of the Daresbury Synchrotron Radiation Laboratory.

A small correlated roughness along q_z (normal to the surface) was derived from the specular and off specular reflectivities implying that the height fluctuation at each surface is mainly randomly distributed. The theoretical analysis of the diffraction profile along q_z showed that there must be an interfacial phase, with a thickness of about 2nm, between the InSb substrate and CdTe buffer layer. The peak widths at both the half maximum and one twentieth of the maximum of transverse scans along the q_y direction at different q_z positions, at which satellite peaks are located near 004, had no systematic variation for different satellite orders. The transverse scan peaks were narrow, being about 17 and 55 arcsecs wide at half maximum and at one twentieth maximum, respectively. These measurements imply, in agreement with the reflectivity results, that the interfaces are very flat. The satellite peak widths along q_z increase from 35 arcsecs for +2 and 85 arcsecs for -1 as q_z moves to the lower satellite orders. Peak splitting can be clearly seen at the -3 satellite peak. These results imply that disturbances or oscillations in the repeated layer parameters of CdMnTe/CdTe had occurred during growth.

APPLICATION OF MULTIPLE DIFFRACTOMETRY FOR DETERMINATION OF CRYSTAL STRAINS.

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The Renninger effect involved in detoured excitation of reflections, forbidden by structure or polarization is only observed at multiwave diffraction. The given effect underlies the majority of methods of a multiple diffractometry [1].

General statements of a multiple diffraction in crystal, containing insignificant deformations ($\Delta a/a \leq 10^{-5}$) are used to receive the quantitative information about crystal strain from X-Ray diffractometry data. The variation of angular distance between multiple maxima corresponding to input and output of the same reciprocal unit on reflection sphere are investigated by the numerical modeling for perfect and deformed crystals [2]. For the analysis the most sensitive to the deformations multiple maxima of intensity are chosen. For the determination of variation of lattice periods value and angles between corresponding planes a system of six nonlinear equations has been solved.

For a number of epitaxial structures $Cd_xHg_{1-x}Te/CdTe$ the analysis of experimental multiple diffraction patterns, received in $CuK_{\alpha 1}$ - radiation is carried out by developed algorithm at basic reflections (222) and (600). Over angular displacements of spectral and deformational sensitive maxima, parameters of discrepancy are found and components of a deformation tensor are calculated.

It should be noted, that the given algorithm enables to determine strain fields in any crystalline structure, in epitaxial systems and at thin film interfaces, to determine parameters of discrepancy in multilayer systems, as well as to find a mutual arrangement of atoms and molecules in the elementary cell of a crystal.

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2. M.D.Raransky, I.M.Fodchuk, V.N.Sergeev, A.G.Gimchinsky, M.D.Borcha//*Metallophyzica,*-1993.*Sov. Phys.*-v.15,#2.-p.80-83.

DIFFRACTION PEAK PROFILES FROM MISFIT DISLOCATIONS IN HETEROEPITAXIAL STRUCTURES

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The misfit dislocations arising on stress relaxation in heteroepitaxial systems are the main source of broadening of the diffraction peaks in these systems. Calculations of peak profiles will be presented, paying special attention to correlations between dislocations due to elastic interactions between them. The analysis is based on the general approach by Krivoglaz [1]. The presence of a free surface essentially modifies the elastic field of the misfit dislocation: the short-range field corresponds to that of a dislocation in an infinite medium whereas the long-range field is similar to that of a dislocation dipole due to image forces.

It can be expected that the misfit dislocations are located at random, but not independently, since elastic interactions between dislocations force them to take low-energy states. Recent analysis of elastically interacting dislocations in a two-dimensional solid [2] demonstrated that correlations between dislocations essentially influence the profiles of the diffraction peaks. The interaction energy $\mathcal{H}$ depends on the distance between dislocations, $\mathcal{H} = \Sigma\hat{\mathcal{K}}(\boldsymbol{r}-\boldsymbol{r}')\boldsymbol{b}(\boldsymbol{r})\boldsymbol{b}(\boldsymbol{r}')$, where $\boldsymbol{b}(\boldsymbol{r})$ is the Burgers vector of the dislocation at the site $\boldsymbol{r}$. The probability of a given dislocation distribution is expected to be proportional to $\exp(-\mathcal{H}/kT)$. Then, proceeding to Fourier transform and applying the equipartition theorem, one has $< \boldsymbol{b}(\boldsymbol{q})\boldsymbol{b}(-\boldsymbol{q}) >= kT\,\hat{\mathcal{K}}^{-1}(\boldsymbol{q})$. The intensity distributions will be calculated for different effective temperatures T and compared with the observed diffraction patterns. The misfit dislocations cause non-uniform distortions of the top surface of the heterostructure. Correlations of the distortions will be determined.

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[2] I. R. Peterson and V. M. Kaganer. *Phys. Rev. Lett.* **73**, 102, 1994.

X-RAY INVESTIGATION OF STRAIN COMPENSATED GaAs:C/AlAs:C DISTRIBUTED BRAGG REFLECTORS

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Abstract

We present an application of high resolution X-ray diffraction, X-ray reflectivity and X-ray topography to investigate a subject of technological importance, the strain compensation in distributed Bragg reflectors (DBR). DBRs form part of complex optoelectronic devices such as vertical cavity surface emitting lasers, microresonators and saturable absorbers. In order to obtain a high optical reflectivity, the DBR must have a large number of quarter-wavelength pairs and hence be of a considerable thickness. This fact together with the difference in the lattice parameters between the constituents materials results in the degradation of the structure by the generation of misfit dislocations. We use carbon as a p-type dopant to produce highly doped DBRs, which are at the same time strain compensated. We apply X-ray scattering methods to determine the best growth conditions, the effectiveness of the strain compensation, the onset of relaxation and the overall structural quality of the DBRs. Our work shows that strain compensated GaAs:C/AlAs:C distributed Bragg reflectors can be grown by solid source molecular beam epitaxy using carbon doping densities up to about 2×10^{20} cm^{-3}. The residual strain with respect to the GaAs substrate can be smaller than 1×10^{-4}. This results in a large increase of the critical thickness of the carbon doped DBRs in comparison with the undoped or Be doped DBRs. We demonstrate that simulations of the X-ray diffraction patterns are essential in order to determine the chemical profile as well as the structural parameters of the GaAs:C and AlAs:C layers with high accuracy. We find that the effective incorporation of carbon on lattice sites in AlAs:C is twice as large as in GaAs:C using the same incident carbon flux.

KINETIC PROPERTIES OF DISLOCATIONS IN SEMICONDUCTORS REVEALED BY X-RAY TOPOGRAPHY

Koji Sumino
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Dislocations often give harmful effects on the performance of semiconductor devices. Plastic deformation of semiconductor wafers which occurs during thermal processing also results in decrease in the production yield of devices. Thus, a great deal of effort has been devoted to grow elemental or compound semiconductor crystals free from dislocations and also to suppress the activity of dislocations during device production processing. The success of such attempts is achieved most efficiently on the basis of the kinetic properties of dislocations in this kind of materials.

Dislocations are induced in a semiconductor crystal when it is under stress at high temperature. The greatest advantage of X-ray topography (XRT) is to allow us to observe dislocation-related phenomena in bulky specimens. Parameters controlling such kinetic phenomena are determined most reliably and quantitatively by in situ XRT. Limitations in applying XRT mostly come from its low resolution and low magnification. Usually some complementary experimental techniques, such as transmission electron microscopy (TEM) and etch pitting, are needed together with XRT to reveal whole aspects of any dislocation-related phenomenon.

This paper gives a rather extensive review on kinetic properties of dislocations in semiconductors clarified by author's group. Emphases are laid on the kinetic properties of dislocations in this kind of materials which could be clarified only by means of XRT and not by other experimental techniques and also on the phenomena that were originally found by XRT but stimulated more detailed research by other experimental techniques. Topics covered are as follows :

1. Dislocation generation ; XRT first showed that dislocations were generated at some surface flaws. The nature of such flaws were later clarified by TEM.
2. Dislocation multiplication ; XRT revealed two basic processes for dislocation multiplication and allowed the formulation of the processes.
3. Dislocation motion ; Dislocation velocity in Si was most accurately measured by XRT. XRT also first demonstrated the dragging effect of oxygen impurities on dislocation motion.
4. Dislocation locking ; XRT first clarified pinning process of dislocations due to oxygen impurities in Si. It led to the unique study of dislocation immobilization phenomena in GaAs and InP by the etching technique.

GRAZING INCIDENCE DIFFRACTION TOPOGRAPHY: THEORETICAL AND EXPERIMENTAL INVESTIGATIONS OF DISLOCATION IMAGES

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The application of grazing incidence diffraction topography (GIDT) is of a great interest since it combines the defect imaging possibilities of the conventional Bragg case topography technique with the shallow penetration depth of the X–rays at small glancing incidence angle. However, investigations of the defect contrast in single crystals and epitaxial layers under GIDT conditions have been incomplete in that no defect data could be evaluated from the observed contrast [1,2] because known theories are not able to describe the contrast at grazing incidence conditions.

In this work we present an approach which describes the GIDT contrast based on a semi–kinematical theory and it exploits the Takagi–Taupin equations with boundary conditions, that take into account the effect of specular reflection. This theory makes use of the Green function method [3] in order to solve the problem analytically.

Images of straight dislocations, which outcrop at the front crystal surface are calculated numerically. In the theoretical derivation, the effect of a free crystal surface on the elastic displacement field of such threading dislocations is taken into account. As an example, computer simulations for threading dislocations in GaAs are analysed for different reflections under GIDT conditions. The theoretical images are compared with experimental results.

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SYNCHROTRON X-RAY TOPOGRAPHIC STUDY OF Si WAFERS AND DEVICE STRUCTURES FOR ADVANCED 0.25μm AND 0.35μm CMOS TECHNOLOGY

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The control and characterisation of wafer defect/dislocation and strain distributions is of crucial importance for the development of future ULSI technologies. One of the most important emerging technologies is the 0.25μm CMOS process to be utilised in advanced logic circuits. Synchrotron large-area, section, Laue back-reflection and grazing-incidence topographic techniques were applied to 200mm p-Si wafers which underwent various advanced CMOS processing steps. Two main process steps were examined: (i) the deposition of epitaxial n- and p-Si on the 200mm substrates and (ii) the definition of "box" isolation structures (shallow trenches in the substrate normally filled with deposited oxide). Both Arsenic-doped (doping density $2x10^{16}$ cm^{-3}) and Boron-doped (doping density $1x10^{15}$ cm^{-3}) Si epitaxial layers of thicknesses varying from 4μm to 2μm were deposited upon CZ p-type Si substrates (where the doping was either $1x10^{19}$ Boron cm^{-3}, or $1x10^{15}$ Boron cm^{-3}). Section topographs reveal that the epitaxial layers are under severe stresses. The epilayers are clearly visible, though there is a clear trend to a reduction in stress imposed on the underlying substrate as the epilayer becomes thinner. In addition, severe distortion of Pendellosung fringing indicates that these stresses are not distributed evenly across the wafer. This built-in stress is most likely due to the fact that the dopant densities, distributions and species (in certain cases) in the epilayer is different from the substrate material, leading to slight lattice mismatches at the interface.This effect is particularly evident in situations where the doping densities are radically different between the substrate and epi-layer. Laue back-reflection topographs verify that the quality of the epilayer-substrate interface improved as the thickness of the epilayer is reduced. Cellular dislocation structures, of the order of a few hundred μm in circumference, have been observed in the more stressed samples. These structures are remarkably reminiscent of what experimenters have observed in LEC GaAs substrates, with relatively dislocation-free regions surrounded by walls containing many dislocations. The structures tend to disappear as the epilayer thickness (and hence stress) is reduced. These results are also confirmed by grazing-incidence topography, where it is seen that the density of point defects on the epilayer surface is reduced as epilayer thickness is reduced. Finally, device test structures are also examined and it is shown that the above SXRT techniques can be usefully applied to process characterisation even for 0.25μm technologies, as a means of evaluating overlayer stress homogeneity and defect generation in and around etched trenches and fine linewidth overlayers.

AUTOMATED DEFECT IDENTIFICATION AND DETECTION IN DOUBLE AXIS X-RAY TOPOGRAPHS USING IMAGE PROCESSING TECHNIQUES

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ABSTRACT

The application of X-ray topography for the characterisation of defects in semiconductor materials has been well established[1]. Double axis topography using laboratory sources has the advantages that besides being cost effective, it enables long range strains to be mapped. The technique has been exploited to study misfit dislocations responsible for the relaxation of strained epitaxial layers[2]. However, data collection, analysis and interpretation can be quite slow. The paper describes the use of image processing techniques to enhance double axis X-ray topographic images and thus enable a fast and reliable identification and estimation of line and other types of defects in semiconductor wafers.

Algorithms were developed and implemented in Windows Visual C^{++}. The image enhancement algorithms combined the steps of mean filtering, erosion, dilation, Laplacian edge, Sobel edge enhancement procedures applied sequentially. The subsequent crucial step was to segment the image by thresholding, to obtained a binarized image. A defect dependent threshold level selection was determined to be an appropriate method for intended thresholding and was accordingly developed. Line count and blob count algorithms were then developed to detect and estimate defects in the images in the form of lines and blobs.

X-ray topographs of a number of Si and InP wafers were obtained by using Bede High Resolution Double Axis Diffractometer and High Resolution X-ray TV Imager, as well as procured from other sources. The result of applying the algorithms developed, to these raw images indicate that the line and blob count estimates are acceptable for topographs with low density of line defects and other types of defects. For badly dislocated samples, a preprocessing of the image before extracting any information was found crucial. In the case of line count algorithm applied to such images, all other types of defects were removed in the preprocessing, which was found to result in the loss of some information. Clustering of lines, a common feature in heavily dislocated lines, were also found to lead to slightly erroneous results.

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Interference Effects in Bragg-Case Synchrotron Section Topography of Elastically Bent Silicon Implanted Crystals

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White beam synchrotron section topography was applied to a silicon crystal implanted with 4.8 MeV α-particles elastically bent with the radius of curvature close to 100 m. A number of section patterns corresponding to different zero-layer and skew reflections was analysed. It was found that the section pattern in the bent sample was drastically different from those observed in a flat sample which were discussed in [1]. The difference consists in the occurrence of the sets of additional interference fringes covering a long distance up to several millimeters behind the main diffraction maximum. It was possible to reproduce the characteristics of the fringes both in the implanted and non implanted region by numerical integration of the Takagi-Taupin equation. The present interference effects were also studied by visualization of intensity distribution in the plane of diffraction using the numerical integration.

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X-RAY TOPOGRAPHIC INVESTIGATION OF GROWTH DEFECTS IN BISMUTH GERMANIUM OXIDE CRYSTALS

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The piezoelectric and optoelectronic bismuth germanium oxide crystals (BGO) have been characterized by synchrotron X-ray topography. This material, currently obtained by the pulling crystal method or Czochralski's method, usually contains strains due to different growth defects associated with the kind of crystal-melt interface. Defects arrangement and generation are discussed in order to control or eliminate them.

Due to the high absorption coefficient of BGO crystals, transmission X-ray topographs were carried out using synchrotron radiation. Crystal slices were cut parallely and perpendiculary to the [001] and [110] pulling axis.

Observed contrasts correspond to grown-in dislocations, bundles and lines, growth striations and facets, mainly. The latest are identified as low index planes. Growth striations follow the shape of crystal-melt interface. Two types of the dislocations can be observed, those from the seed and others from the growth accidents. Their paths are nearly perpendicular to the crystal growth front and they can show refraction from a growth facet to another.

It can be concluded that the growth of BGO crystals with a convex and slightly faceted interface promotes the dislocation divergence and their spread to the crystal edges leading to crystals with high quality central zone.

ENHANCED POSSIBILITIES OF SECTION TOPOGRAPHY AT A THIRD GENERATION SYNCHROTRON RADIATION FACILITIES

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Section topography is a well known basic technique to investigate the defects within a crystal [1]. When working at a third generation synchrotron radiation facility like the ESRF this technique exhibits improved possibilities associated with the high intensity, the small angular size of the source, and the high energy photons present in the beam. These possibilities are illustrated by the following examples:

1) Multiple sections

The typical exposure time of a section topograph at the ESRF is a few seconds. Once the crystal and slit are adjusted it is easy, by using a translation, to record several sections, separated enough not to be superimposed and close enough to be able to follow a given defect from one section to the next one. This technique which can provide simultaneously the information of section and projection topographs, was used for instance to investigate the defects created by the application of an electric field in α-$LiIO_3$ crystals.

2) Weak misorientations

The small size of the source allows good geometrical resolution to be retained even when setting the detector far (~ 1 m) from the crystal. We took advantage of this feature to observe weakly misoriented (~ 1 arc second) surface layers, such as a hydrogen implanted layer in a $LiNbO_3$ crystal.

3) Thick or heavy crystals

The high energy photons allow penetration of thick or heavy samples. Kato's fringes have been observed in a 1 cm thick silicon crystal and bulky (several cm^3) as-grown crystals could be characterised prior to cutting [2].

4) Real time section topography

The high beam intensity and, therefore, the short exposure times allow the real time study of the magnetic phase transition in MnP, near the triple point at 47 K. We used a 15 µm wide slit consisting of two pieces of tantalum, each 0.5 mm wide, and could observe, using a CCD camera not only a small section of the sample (section topograph), but also the region outside the slit (projection topograph). This makes it easier to follow the evolution of the coexisting phases and interpret the topographs. We also studied through section topographs the incommensurate phase of quartz at around 848 K. We could follow in real time (in spite of their weak intensities) the position of the satellites during the phase transition, and, in this way, relate the phase transition temperatures to the local crystalline quality [3].

5) Monochromatic sections

The ESRF monochromatic beams appear, for most of the investigated crystals, as "quasi-plane waves". It is, in addition, possible to use the "weak beam" technique, which allows to image the very distorted parts of the scanned volume. Monochromatic sections were used to study a highly oriented pyrolitic graphite sample, and for the first time directly image the small misoriented mosaic blocks, and establish a correlation between their orientations and the growth process.

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SYNCHROTRON WHITE BEAM X-RAY TOPOGRAPHIC INVESTIGATION OF THE ANNEALING BEHAVIOUR OF QUASICRYSTALS DEFECTS.

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Quasicrystal defects are poorly known. Defects like those in crystals, such as dislocations, stacking fault, subgrain boundaries ... have been identified, recently, in quasicrystal grains, but the strain field around them is still unclear. Despite it has been proposed, theoretically, that the strain field around quasicrystal dislocations can be considered as split into two components referenced in two subspaces, the physical one $E_{//}$ (phonon strain) and the perpendicular one $E_{\perp}$ (phason strain), which are part of an hyper periodic space inside of which the dislocations are created by a Voltera process, it remains to determine the extent of the strain field in each subspaces and therefore how these defects can move.

The aim of this paper is to report the results of the 1rst "in situ" and Real Time Synchrotron White Beam X-Ray Topographic investigation of the annealing behaviour of quasicrystal defects. It has been carried out in AlPdMn icosahedric grains and it will be shown that these defects have an unsual annealing behaviour which can be related to the theoretical predictions concerning their strain field.

X-RAY DIFFRACTION ASSESSMENT OF ROUGHNESS AND ATOMIC ORDERING AT SURFACES

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Probing the quality of a crystal surface in terms of smoothness and long-range order has become a major issue for the design of optimized devices in the nanometer range. Among the possible methods, Surface X-ray Diffraction is a most efficient tool when coupled to the intense synchrotron radiation sources. Surface sensitivity is achieved in the grazing incidence geometry whereas buried interfaces can be reached by tuning the penetration depth with the glancing angle. Due to the small size of the diffracting object, surface diffraction phenomena are well interpreted in the framework of the single scattering kinematical theory. Examples of applications to reconstructed surface structures and phase transitions will be presented. A special attention will be given to the detection of surface defects such as antiphase boundaries, stoichiometry variations on surface steps, by specific features in the diffraction pattern. The rodlike shape of the intensity distribution in semi-infinite and quasi 2D systems will be exploited to derive information on the surface roughness and structure.

INVESTIGATION OF THIN QUANTUM WELLS BY X-RAY DIFFRACTOMETRY, REFLECTIVITY AND DIFFUSE SCATTERING

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Thin GaAs single quantum wells of a thickness near 4.5 nm with barriers consisting of AlAs/GaAs short period superlattices (10 ... 50 periods) grown by molecular beam epitaxy on GaAs (001) are investigated by means of high resolution diffractometry, reflectivity and diffuse scattering.

The mean thickness of the quantum well can be determined with an accuracy of at least 0.5 nm and the average thickness of the superlattice layers are determined with an accuracy of 0.01 nm by comparison of the results of diffractometry in the (002) and (004) reflections with dynamical simulations. The average thickness of „graded" interface layers is determined by X-ray reflectivity measurements to be near 0.3 nm. These graded layers at the interfaces are taken into account in the simulations. The grading may be connected with Gallium segregation and with interface roughness. The main features of the diffraction pattern are explained with a kinematical model.

The roughness of the interface layers is characterized in more detail by X-ray diffuse scattering. Experimental results are compared to simulations in the distorted wave Born approximation. The roughness over larger areas is highly correlated whereas the vertical correlation lengths are reduced for the shorter spatial frequencies. Typical values for the correlation lengths are: vertically 0.02...0.2 μm, laterally 0.1...1 μm. For comparison the surface roughness is measured by atomic force microscopy. The interface roughness of the quantum well and the superlattice barriers are characterized by cathodoluminescence in the scanning electron microscope via the line shift connected with different layer thicknesses taking into account the averaging due to the limited resolution of the microscope. The values for the average roughness agree well between the different methods. Further work is needed to interprete the values of the correlation lengths.

MODELS OF THE CORRELATED INTERFACIAL ROUGHNESS IN MULTILAYERS AND DIFFUSE X-RAY SCATTERING

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The wide application of multilayers synthetic microctructures determines the necessity of their structure control. X-ray coherent (specular) and diffuse (non-specular) scattering yield the information regarding the detailed morphology of such structures [1, 2]. But the existence of correlated interfacial roughness gives rise to the set of different parameters which treatment is important for the complete picture of the interfaces in multilayer structures [3].

The model of interfacial roughness which can be perfectly correlated, uncorrelated or partially correlated was built. It takes into account of both the long-wavelength roughness and the short-wavelength one. The degree of the correlation is determined by the vertical perfect, the vertical partial and the lateral correlation length for the each type of roughness. According to this model the specular component of the scattering contains the information about the vertical perfect correlation length and the form of the dependence of the mean-square roughness from the distance between the interfaces. The rest important parameters can be derived from the diffuse component

The second model describes interfacial roughness in the reciprocal space. It gives the wavelength of interfacial roughness and the magnitude of the roughness correlation.

The computer simulation of the scattered intensity at the high resolution triple crystal diffractometry method based on both phenomenological models makes it possible to extract the parameters of correlation and roughness in case of the different number of periodical layers in multilayers. For a single rough interface the effect of multicrystal convolution on a true distribution of X-ray Yoneda diffuse scattering is discussed.

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INTERFACIAL ROUGHNESS CORRELATION IN SUPERLATTICES STUDIED BY X-RAY DIFFUSE SCATTERING

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The formation of interfacial roughness is fundamental for the understanding of basic characteristics of the growth process. The mean amplitude of roughness can be evaluated from x-ray reflectivity. Due to interfacial roughness the intensity of the coherent superlattice Bragg peaks is strongly damped with increasing reflection order. Lateral information on roughness, in particular the lateral correlation length $\xi_{\|}$ and the fractal dimensionality h, is only accessible when diffuse scattering is investigated [1]. Recently, it has been shown that perfect roughness correlation between different interfaces gives rise to intensity concentration in resonant diffuse scattering (RDS) sheets [2,3].

We have investigated a $(GaAs/AlAs)_n$ superlattice (n = 20) grown on a GaAs (001) substrate by molecular beam epitaxy. High resolution x-ray diffractometry has shown that all layers are grown pseudomorphically onto the substrate. X-ray reflectivity and x-ray diffuse scattering have been measured with a two circle goniometer installed at a 12 kW rotating anode generator (Cu K_α radiation). Additional measurements were performed at wiggler station W1 at HASYLAB using an x-ray wavelength of $\lambda = 0.1395$ nm. The superlattice period has been determined to 22.7 nm with layer thicknesses of 7.3 nm and 15.4 nm for the GaAs and the AlAs sublayers, respectively, and for the thickness of the top oxide layer we evaluated a value of 2 nm. The diffuse scattering has been measured by reciprocal space mapping using a linear position sensitive detector. We observe very strong RDS sheets indicating strongly correlated interfaces. We measured RDS up to incident and exit glancing angles of $\alpha_{i/f} = 4°$ which represents a maximum reflection order of N = 22. In a detailed quantitative analysis we will discuss different roughness models by Ming et al. [4] and by Spiller et al. [5]. The latter one takes into account that interfaces are formed successively one after another. This is expressed by a vertical correlation length which depends on the spatial frequency of roughness.

Also Bragg diffraction peaks in the diffuse scattering have been predicted [2,3].This phenomenon appears when either (i) the incident or (ii) the scattered wave fulfills the Bragg diffraction conditions and can be explained using the concept of *Umweganregung*. We have measured these Bragg peaks and will discuss their behavior with regard to our theoretical approach [3].

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X-RAY DIFFRACTION STUDY ON THE CORRELATION BETWEEN ORDERED DOMAINS SIZE AND ORDERING DEGREE IN InGaP/GaAs ALLOY LAYERS

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Long-range ordering in III-V ternary alloy semiconductors deserves careful investigation since it is related with significant changes in the electrical and optical properties and provides an additional degree of freedom in band engineering. Therefore, the understanding of nature and origin of the deviation from randomness is fundamental for the control of the device performances.

Nominally lattice matched InGaP/GaAs heteroepitaxial layers, grown under proper conditions, exhibit long-range order in the cationic sublattice along the <111> directions.

The effect has been mainly studied by High Resolution X-Ray Diffraction (HRXRD) comparing the results with the findings of complementary tecniques like Transmission Electron Microscopy (TEM) and Photoluminecence (PL).

Undoped heteroepitaxial layers of InGaP with constant thickness have been grown lattice matched by Methalorganic Chemical Vapour Deposition on GaAs substrates with miscut angles of 0°, 2°, 6° with respect to the (001) direction; moreover n and p doped samples have been grown on 2° off substrates.The X-ray diffraction mesurements were performed both using a Philips diffractometer equipped with a two-crystal four-reflection monochromator (Ge, 220) and the Cu $K\alpha_1$ (λ=1.54 Å) as radiation, and a high resolution four circles diffractometer at LURE syncrotron radiation center (Orsay, Paris) with a fixed wavelenght λ=1.38 Å (absorption treshold of Ni). The examined InGaP layers spontaneously order in CuPt-like domains presenting (-111) and (1-11) variants. The doubling of periodicity has been revealed both by TEM and HRXRD by the appearence of extra reflections at (-1/2 1/2 5/2) and (1/2 -1/2 5/2) positions, while (1/2 1/2 5/2) and (-1/2 -1/2 5/2) peaks were not found. The Full Width at Half Maximum (FWHM) of the (1/2 -1/2 5/2) diffraction peak has been found to decrease as the miscut angle increases corresponding to an increase of the size of the (1-11) ordered domains while, the (-111) variant gradually disappears.

Moreover, the FWHM and the integrated intensity of the four reflections (±3/2 ±3/2 3/2) were mesured. According to previous results, the cationic ordering has been evidenced only on the (1-11) and (-111) planes and the domains size increases when the angle of the ordered plane decreases with respect to the surface, while for the integrated intensity the opposite occurs. This last finding is a clear evidence of the decrease of the ordering degree in the bigger domains. Up to date, the degree of order within the domains has not been investigated and the results will be discussed on the basis of the correlation between domains size and ordering degree.

Finally, the doping level effects (Si [n] and Zn [p]) on the cationic ordering will be analysed.

X-RAY SCATTERING FROM THIN ORGANIC FILMS AND MULTILAYERS

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Abstract

Multilayers from fatty acid salt molecules provide a model system for the study of the 3D order of molecules within lamellar organic films. X-ray scattering techniques are widely applied to evaluate the vertical stacking of the film. The traditional methods of data interpretation fail if the defect structure of the sample used is not taken into account. For multilayers prepared from fatty acid salt molecules by means of the Langmuir-Blodgett technique the 3D domains are the most important "defects". They can be visualized by AFM on the sample surface, but their existence cannot be proved in the bulk of the sample. Where as the X-ray specular reflectivity (XSR) probes the vertical stacking, the X-ray grazing incidence diffraction (GID) gives information about the in-plane arrangement of molecules within these domains. Additional information can be obtained from X-ray diffuse scattering (XDS) experiments. But GID and XDS measure different lateral lengths. The angular width of in-plane rocking curves recorded by GID is explained by the coherence of the crystalline lattice which fulfil the in-plane Bragg condition. Using a highly collimated synchrotron beam an averaging size of several 100Å is evaluated which changes as a function of deposited monolayers. XDS scans exhibit an increase of the angular width of incoherent scattered intensity as a function of the order of small angle Bragg peaks. It has been shown recently that its functional dependence contains information about the in-plane correlation length of the average electron density if the interface roughness is known. This correlation length is in the order of about 2500Å. Because the GID and XDS results differ by at least one order of magnitude, they reflect different scattering aggregates. Whereas the XDS proves the size of the macrodomains the GID measures the single-crystalline microdomains which appear within the macrodomains. Furthermore, the lateral coherence length of the probing X-ray reveals as an additional crucial factor for the evaluation of different types of imperfections.

INTERFACE STUDY OF THE W/Si MULTILAYERS WITH INCREASING NUMBER OF PERIODS

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We have studied the interface evolution across a multilayer (ML) on a series of the W/Si MLs with increasing number of periods by the X-ray reflectivity and diffuse scattering measurements at grazing incidence. These MLs are used as X-UV optics elements. The number and quality of the interfaces plays a decisive role in achieving their optimum performance. A proper interface description is of crucial importance to interpret duly the experimental results. A closer inspection of this point, taking into account various possibilities, is another goal of our study.

The samples were prepared by the UHV electron-beam deposition on oxidized Si (100) wafers with a 0.5 μm thick SiO_2 passivation layer starting with Si, the basic bilayer (5nmSi/1nmW) being repeated N=3, 6, 9, and 15 times. The measurements were performed on the Stoe high-resolution diffractometer equipped with a double-crystal GaAs monochromator in +/- arrangement working at 400 reflection and cut under 3° with respect to the (100) plane using $CuK_{\alpha 1}$ radiation. A compromise between the spectral purity and intensity of the primary beam is reached in this way. The internal structure of the individual layers was found to be amorphous by a conventional Bragg-Brentano diffractometer with a curved graphite monochromator in the secondary beam.

The mapping of the intensity distribution in the reciprocal space was done by the specular, 2θ, and ω scans close to the total external reflection. The results were evaluated within the Fresnel optical approach and the distorted-wave Born approximation. The reflectivity on the 1st ML Bragg maximum increases from 20% to 55% on increasing N from 6 to 15. A model of vertically correlated interfaces [1] was successfully applied for all the samples giving nearly the same substrate/passivation layer interface roughness of 0.60-0.68 nm and a roughness increment across ML of 0.01 nm, the lateral correlation length being 10 nm. The interfaces exhibit fractal behaviour up to N=9 with fractal dimension of D=2.8-2.85 while for N=15 their fractal and topological dimensions are the same. It was found that sensitivity of the simulation results to the change of the fractal behaviour in the interval D=2-2.5 is weak.

The appearance and evolution of the ML kinematic and dynamic scattering effects with increasing N, as manifested in the non-specular scans, is discussed. Other models of the vertical interface conformity are tested and compared, namely a phenomenological one with an exponential decay of the cross-correlation function across ML and that described in [2], supporting the conclusion about the correlated interfaces in our samples.

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THE MEASUREMENT OF ELEMENTAL DEPTH DISTRIBUTIONS IN THE SURFACE LAYERS OF GLASSES USING COMBINED X-RAY REFLECTIVITY AND FLUORESCENCE

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The optical and physical properties of glasses can be significantly modified by the presence in the surface and near surface of a variety of elements. These may be present either intentionally, as in the case of thin films grown on the surface, or as a contaminant introduced as part of the manufacturing process. The concentration distribution, as a function of depth below the surface, is an important factor in determining the properties, both for elemental concentration variation in the surface layer of a bulk specimen and for layered specimens, where the major interest is often in the concentration gradient at the film/bulk interface.

X-ray penetration at grazing (near critical) angle incidence is dependent on both the incidence angle and the surface/near surface electron density, allowing the penetration to be controlled in the range ~1nm to ~1μm. Elemental depth distribution information can thus be obtained from the combined measurement of X-ray reflectivity and X-ray fluorescence as a function of the X-ray incidence angle. Measurements were made using a Bede GXR1 reflectometer, modified by the addition of an Ortec Si(Li) energy dispersive detector positioned immediately above the specimen. Cu Kα radiation was used, allowing detection of elements up to $Z = 27$ by K line fluorescence and up to $Z \equiv 65$ by L line fluorescence.

Selected experimental glass specimens made by Pilkington Technology Management Ltd. were studied. Measurements were made of the concentration distribution of Fe and Sn, which, at varying concentration levels, can be present. These two elements illustrate the upper and lower sensitivity levels that may be achieved in this range of elements. The fluorescence signal from Fe in the specimens studied gives a well discriminated pair of K line peaks whereas the L lines of the Sn fluorescence spectrum have a substantial overlap with the K lines from Ca, a major constituent of these glasses, thus making signal deconvolution necessary. Comparisons are presented of the experimental data with simulations of the fluorescence intensity dependence on incidence angle for particular assumed surface distributions. The sensitivity to both concentration and depth is discussed for these differing cases.

Glancing Incidence Resonance Enhanced X-rays (GIREX) : a tool for structural studies and for X-ray wave guide production.

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It is well known that x-rays striking solid surfaces under a certain critical angle α_c are totally reflected [1]. Then an incident coherent plane wave will produce a standing wave field above the surface [2], due to the interference between the incoming and the reflected beams. The standing wave periodicity D depends on the grazing incident angle a and on the wavelength λ: D = l/2 sin(a).
If a thin film with a smaller critical angle is deposited onto the solid surface, then the incoming beam penetrates the film but it will be totally reflected from the underlying surface, with the consequent formation of a standing wave field in and above the film. If the film thickness is properly chosen, standing wave nodes take place at the interfaces air-film and film-surface. Under these conditions a considerable enhancement of the e.m. field intensity in the film can occur [3,4], resulting in reflectivity spectra displaying deep minima. This happens any time the number of standing waves between the interfaces is an integer (resonance orders).

Two main applications of the GIREX technique are envisaged:
i) structural and compositional study of thin films
ii) construction of a hard X-ray wave guide.

i) The existence in the thin films of standing wave nodes and antinodes gives the possibility to accurately determine (with about 1% accuracy) the position of markers through the measurements of secondary effects (for ex. fluorescence). On the other side the thickness of the film can be determined within 1% through the angular position of the minima in reflectivity. Furthermore, information on interface roughness can as well be obtained. Examples of this kind of study will be presented on structures formed by an amorphous C film and a Ti layer placed at different depth in the film [5].

ii) An x-ray wave-guide composed of a thin C film sandwiched between two Ni layers deposited onto a glass substrate has been constructed and tested at ESRF. A beam with one dimension as small as 100 nm has been evidenced at the end of the wave-guide. Its intensity, direction and divergence has been measured. An efficiency of about 10^{-4} has been obtained, but there are strong hopes, based on theoretical predictions, that it can be significantly improved. Work is in progress to fabricate more efficient wave-guides and to apply them to x-ray microscopy and microfluorescence studies.

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X-RAY TOPOGRAPHIC STUDIES OF ORGANIC AND NON-LINEAR OPTICAL MATERIALS

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The flexibility and non-destructive nature of X-ray topographic techniques are ideally suited to the evaluation and study of large single crystals for both fundamental research and technological applications. The ability to examine large crystal slices or entire crystals is of particular value in the evaluation and optimisation of crystal growth processes. This paper will focus on three examples of the application of a range of X-ray topographic methods to such investigations in the case of non-linear optical (NLO) crystals.

Synchrotron radiation section topography has been applied to the examination of large (up to 2cm thick) organic crystals. Examples taken from the important NLO materials urea and 2-(α-methyl benzylamino)-5-nitropyridine (MBANP) demonstrate the advantages of this totally non-destructive assessment of crystal quality and illustrate the valuable insight into crystal growth history which can be obtained.

Relatively little information is available from X-ray topography concerning the nature of growth induced defects in crystals grown from the melt by the Bridgeman method. This is principally due to the high dislocation densities and substantial lattice strain typical of confinement of the growing crystals in a rigid ampoule. Despite such difficulties, X-ray topography has been used to examine growth defects and crystal quality of crystals of m-nitroaniline (mNA) grown by the Bridgeman method. These studies allow evaluation of growth parameters and their influence on defect density and show that in the case of mNA, remarkably low defect densities can be achieved under optimum growth conditions.

The use of double crystal reflection topography has, in the past, been confined primarily to the examination of highly perfect crystals such as silicon. The versatility and surface sensitivity of the technique, however, offer considerable advantages for the examination of near-surface defects in other, less perfect materials. Double crystal reflection topography, with synchrotron radiation has been used to image defects intersecting the {011} faces of the inorganic NLO material potassium titanyl phosphate (KTP). X-ray images have been combined with differential interference contrast microscopy and optical interferometry to provide valuable information on the crystal growth process

QUASI REAL-TIME WHITE BEAM SYNCHROTRON TOPOGRAPHY STUDY OF DEFORMATION IN COATED Ni_3Al SINGLE CRYSTALS

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White beam synchrotron x-ray topography was used to study the lattice distortion in Ni_3Al single crystals coated with refractory metal multilayers, particularly in the vicinity of Ni_3Al yield region. Stop-action x-ray topographs were taken during *in-situ* tensile loading, with the beam direction normal to the specimen surface and the tensile axis. Two different types of multilayer coatings, namely Mo/W and Ta/W, were applied to the Ni_3Al substrates using magnetron sputtering. These coatings were previously shown to possess simultaneous high strength and toughness. The present work focused on the effect of the multilayer coatings on the mechanical behavior of Ni_3Al substrates. From an analysis of the stress-strain curves and x-ray topographs, the microyield stress of the Ta/W coated samples was determined to be about 50 MPa higher than that of uncoated Ni_3Al. The increase in the microyield stress was due to tensile residual strains in the Ta/W multilayers, exerting compressive strain on the Ni_3Al substrates. The microyield stress for the Mo/W coated samples, on the other hand, was about 50-100 MPa less than that of uncoated Ni_3Al. This was related to the compressive residual strains in the Mo/W multilayers exerting tensile strain on the Ni_3Al substrates. The residual strains were determined from an analysis of diffraction peak positions. The macroscopic yield stress results obtained from the x-ray topography studies will be discussed in relation to the localized slip band structures at the substrate/coating interface using transmission electron microscopy.

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X-Ray Topographic Study of Twins in $Nd_xY_{1-x}Al_3(BO_3)_4$ crystals

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Abstract

Transmission synchrotron radiation topography is applied to investigate the twins in self-frequency-doubling laser crystal $Nd_xY_{1-x}Al_3(BO_3)_4$ (NYAB). It is found that the twin in NYAB is similar to the Brazil twinning in natural quartz, i.e. the twinning is symmetric by reflection in prismatic planes {1120} and its composition planes are the {1011} types. It is known that the Brazil twin contrast originates from the phase difference α suffered by the X-rays on the twin boundaries. Using the atomic coordinates for the NYAB crystal and twinning structures, we have calculated the phase angles θ_H and θ_T for the host crystal and twinning respectively. The calculations show that the phase angles θ_H and θ_T are not always identical for all reflections. The contrast of the transmission synchrotron radiation topographs obtained with different reflections is in agreement with calculations. When the θ_H and θ_T are equal, the twinning is invisible. Otherwise, the twin contrast can be observed.

LAUE SYNCHROTRON TOPOGRAPHIC STUDIES OF PHASE TRANSITIONS IN $KLiSO_4$

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$KLiSO_4$ with a tetrahedral framework structure exhibits a rich temperature polymorphism involving various orientations and disorder of the SO_4- and LiO_4-groups. At least ten phases have been reported between 5K and 989K (melting point) [1]. Most of their crystal structures are still unknown. Extensive X-ray topographic studies of this compound were carried out by Klapper, Hahn & Chung [2] and Jennissen [3].

Phases	I	← 949K →	II	← 708K →	III	208K → ← 243K	IV	← 190K →	V
Space group	$P6_3/mmc$		$Pmc2_1$? $P2_1cn$?		$P6_3$		$P31c$		$P112_1$? Cc?
	paraelectric		ferroelastic		pyroelectric		pyroelectric		

Table I: Partial phase diagram of $KLiSO_4$ (normal pressure) [3]

We have carried out in-situ topographic studies of the dynamics of the phase transitions as well as their phase boundaries and domain formation. $KLiSO_4$ plates cut normal to the main crystallographic directions were examined by white-beam synchrotron topography in between 140K and 980K at the SRS Daresbury, UK. The white-beam technique allows to study lattice tilts without leaving the reflection condition. In addition the intensity is sufficient for good time resolution (typical exposure times: 120 seconds). Topographs were recorded during different stages of the four phase transitions, using heating and cooling chambers with adjustable temperature gradients, from which the movement of the phase boundary through the plate can be traced.

The transition III [$P6_3$] - II [orth.] at 708K generates a twin structure with three orientation states. Due to the lattice tilts of the numerous domains and new defects the topographs appear blurred. During re-transformation a further, not-yet characterized twinning appears. The pseudo-hexagonal domains of phase II vanish during the transition II [orth.] - I [hexag.] at 949K (reduced blurring of the topographs). In both transitions and in both directions the phase boundary follows closely the transition isotherm (no hysteresis).

The transition IV [$P3_1c$] - III [$P6_3$] is sluggish with a hysteresis from 208K to 243K. Upon cooling below 200K the trigonal phase IV transforms into the monoclinic phase V, which is densely twinned with (at least) three orientation states. In contrast to the high-temperature transitions the original (grown-in) twin domains appear again after re-transition into phase III (memory effect).

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STUDY OF THE DIRECTIONAL SOLIDIFICATION OF DILUTE Al-Cu ALLOYS BY IN SITU SYNCHROTRON X-RAY TOPOGRAPHY

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The evolution of characteristic patterns (cells, dendrites) with growth rate is analysed on X-ray topographs taken in situ and in real time during directional solidification of thin samples of a dilute Al-Cu alloy.

Primary spacings λ and tip radii R are measured for cellular patterns and in the beginning of dendritic growth. As the pulling velocity V is increased the typical S-shape for the λ (V) curve is proved valid for thin metallic samples. The primary spacing first decreases, then increases up to slightly beyond the cell-dendrite transition and finally decreases again in the dendritic domain. As a probable consequence of a weak and local wavelength selection both patterns can coexist in the cell-dendrite transitory range. The experimental evolution of primary spacing is compared with corresponding results on bulk samples available from the literature. The main differences appear in the cellular range ; for bulk samples the liquid convection influences the shape parameters and delays the cell-dendrite transition.

The formation and evolution of elastic strain contrasts displayed just beyond the critical growth rate, the movement of dislocations in the intercellular grooves as well as the presence of dislocations inside deep cells in the predendritic regime are discussed.

Study of residual strains in wafer crystals by means of Bragg angle mapping

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Abstract

A high resolution x-ray diffractometer equipped with an accurate X-Y translation stage has been used to obtain the Bragg angle maps of large-area crystal wafers. It is found that the Bragg angle shifts observed by this method in conventional reflection geometries are mainly due to the local crystal curvature rather than to the lattice parameter changes. The same is responsible of the contrast variation due to the diffracted intensity variations in the plane wave x-ray topography technique.

The displacement of the crystal lattice sites with respect to the ideal unperturbed lattice is calculated from the Bragg angle maps. It is demonstrated that such lattice displacement is quantitatively correlated to the wafer dislocation density and to the distribution of the Burgers vectors of the dislocations in the crystal.

Semi insulating Czochraslki grown GaAs wafers with dislocation densities ranging from 10^4 to $10^5 cm^{-2}$ have been analysed by this technique and by double crystal X-ray topography. The dislocation density map evaluated appeared very similar to those determined by topography and etch pit density in this material. Moreover the value of dislocation density found suggested that the majority of dislocations in the wafer have the same Burgers vector component along the growth axis.

The method is proposed as a new tool for characterizing large area wafer crystals.

OBSERVATIONS OF ACOUSTIC VIBRATIONS BY X-RAY STROBOSCOPIC TOPOGRAPHY

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Stroboscopic X-ray topography of 1ns time resolution obtained with the synchrotron radiation (L.U.R.E., Orsay, France) has been used to study different types of acoustic vibrations in different devices. The studies have shown that time-resolved images of the acoustic waves reveal the existence of particular modes which cannot be observed on time-integrated images obtained by conventional methods. Some examples of B.A.W. (Bulk Acoustic Waves) observed with this technique in quartz resonators are given. Furthermore, a theoretical calculation has been carried out to characterize Pendellosung fringes on stroboscopic X-ray section topographs in weakly vibrating crystals (resonators). The agreement between the theoretical simulation and the experimental results demonstrates that for low vibration amplitude range (u_o =7Å), Pendellosung fringes can be related to the vibration amplitudes.
Some observations concerning the variation of dislocations contrasts in fonction of the vibration amplitude have been done and will be also reported.
As in the case of the B.A.W. observations of S.A.W. (Surface Acoustic Waves) were performed using the synchrotron radiation. In this communication we report investigations which have been made to study the energy distribution of these acoustic waves in different devices made with quartz and lithium niobate. A ST quartz slice working at 9.9 MHz was particularly studied and it was observed that the component in the propagation direction presents a very strong interaction with the dislocations. The form of the emitted beam is strongly modified by the dislocations bundles. The energy distribution in other S.A.W. filters and dispersive delay lines using ST quartz or Y-Z niobate has also been studied and will be reported.
High frequency (60 MHz) filters of lithium niobate were also investigated. The lateral variation of the displacement component normal to the surface was analysed as a function of the frequency and of other experimental parameters.An example is given by a filter made with a Y Lithium niobate with a Z propagation working at 60 MHz with a bandwidth of 3 to 4 MHz. This sample was studied in detail variing the experimental conditions (frequency, incidence angle....). It was observed that the beam emitted by the apodized transducer is highly complex and the topographs explain the high insertion losses.
These investigations have permitted to obtain new insights of the B.A.W. and S.A.W. devices and of the interactions between the waves and the crystal defects and plate edges.

PHOTOELECTRONS IN X-RAY STANDING-WAVE TECHNIQUE: POTENTIALITIES IN CRYSTAL SUBSURFACE LAYER INVESTIGATION

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The well known technique of X-ray standing waves (XRSW) is recently receiving increasing attention. This is mainly due to the possibility of localizing adsorbed impurity atoms and, implanted or diffused atoms at small depths in the specimen, by analyzing the fluorescence radiation yield. Unfortunately, till now, the advantages of using another secondary radiation, such as photoelectrons, have not been fully exploited. This was partly due to the experimental complexity of XRSW-photoelectrons and, partly, to data interpretation difficulties. Nevertheless, the small escape depth (0.01 - 0.5 μm) and the correlation between the kinetic energy loss and the electron generation depth, give us a very efficient tool for the investigation of extended layers disturbed by the above mentioned processes.
Moreover, the angular dependence of the photoelectron yield gives the technique a particular sensitivity to extremely thin amorphous surface layers, which are completely "invisible" to conventional X-ray techniques.
In this work, the specific features of some aspects of photoelectron detection and energy analysis in X-ray diffraction experiments will be presented and discussed.
The most important question in the quantitative analysis of the data is the reliability of the values of photoelectron escape depth. Experimental techniques based on XRSW measurements in the inclined-Laue-diffraction geometry and, on the well known Pendellösung effect for the reconstruction of the probability functions of different photoelectron escape depths [1], are presented.
The two main approaches to the characterization of subsurface layers by photoemission techniques are:
- transformation of the photoelectrons energy analysis into a depth-selective analysis of the crystal structure;
- use of XRSW data as additional information in high resolution X-ray diffraction.
The first approach permits the reconstruction of distortion profiles directly from energy-selective data. In the second, XRSW investigations give the phase of diffracted waves on the crystal surface. Then, they can be used to select the most appropriate strain and damage profiles amongst those which are practically equivalent from the point of view of the kinematical diffraction. Typical results from both the experimental approaches will be given and discussed.

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THE ROLE OF MULTIPOLE TERMS IN THE PHOTOELECTRON YIELD STUDIED WITH X-RAY STANDING WAVE METHOD.

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Nowadays the X-ray Standing Wave (XSW) Method has proved itself to be a powerful tool for structure investigations such as the position of adsorbates on the surface or of impurities in the bulk of the crystal. However, this method could be effectively utilized for the investigation of the inelastic process itself. The photoelectron absorption is the leading inelastic process for X-ray propagation in crystals with energies E=1÷25 *keV*. So, for structure analysis, as well as for investigation of inelastic scattering, it's essential to understand the details of photoelectron process itself.

One of the problems that has to be taken into account while the XSW analysis is: how essential are the multipole expansion terms in the photoelectron yield process. It is well known from the theory of photoeffect that the dipole term gives the main contribution to the photon decay process. However, as it was shown by H.Wagenfeld [1], even for the medium X-ray energies the contribution of the quadrupole terms to the total photoelectric cross section is not negligible and for different elements could be of the order of ~1÷6% It's clear that this contribution is becoming more essential for shorter wavelengths.

In the paper the theory of photoelectron yield taking into account the multipole expansion terms is revised. Each multipole term has it's specific angular dependence. Two different types of experiment are analyzed: integral photoelectron yield and angular resolved photoelectron yield. This results are applied to XSW method. An accurate analysis taking into account multipole terms is performed. Different experiments are proposed for the direct determination of multipole terms directly from XSW analysis.

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STRUCTURAL CHARACTERIZATION OF BURIED INTERFACES BY X-RAY REFLECTIVITY AND X-RAY STANDING WAVES

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Non destructive structural characterization of buried interfaces can be accomplished by various interface sensitive x-ray techniques. This talk will stress the advantages of combining the results from Crystal Truncation Rods (CTR), x-ray reflectivity at grazing incidence (GIXR), and x-ray standing waves (XSW). All three methods give detailed and complementary information. For example by XSW the lattice position of different atomic species at the interface can be determined whereas CTR gives the average position of the lattice planes and in addition the values of the interface roughness and the layer thickness.

The combination of these methods was applied to δ layers. In the ideal case these buried layers should only consist of one smooth layer of e.g. Ge-atoms inside a Si host lattice. This system is interesting, both from the scientific and the technical point of view as a model system for layers of monolayer thickness[1]. Here samples of 1-6 Monolayer Ge and Sb on Si(100) have been prepared by Molecular Beam Epitaxy (MBE), Solid Phase Epitaxy (SPE), and Surfactant Mediated Epitaxy (SME) at various growth temperatures ranging from room temperature to 600°C. These layers have been capped by ≈100 Å Si.

In summary the experiments proof that Ge δ layers of high quality can be prepared by MBE[2]. The quality even improves when using SPE and SME. The residual roughness of the δ layers can be attributed to intermixing and island formation at higher coverages[3]. Sb δ layers could only be produced with larger intermixing and interface roughness than in the case of Ge.

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X-RAY STANDING WAVE FIELDS CLOSE TO BRAGG-BRAGG MULTIPLE DIFFRACTION REGION

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The phase sensitivity of multiple diffraction has been observed long ago, and almost immediately it was suggested to use this phenomena to study the crystal structure. However in the closest vicinity of the exact Bragg multiple diffraction conditions it is impossible to unambiguously interpret the experimental results because the interaction of the wavefields corresponding to different reflection is of essentially nonlinear character.

The linearity is achieved when the crystal is rotated by hundreds of arc sec from the exact multiple diffraction conditions[1]. In this region, the rocking curves for the reflection involved in the diffraction bear information on the phase of the structure amplitude of the finite-thickness subsurface layer; the characterized layer is the thinner, the greater angular displacement crystal from exact Bragg position. The phase-sensitive regions of the rocking curve correspond to the situation when one of the reflections is strong and should be described dynamically, whereas the reflectivities corresponding to all other reflections are low (because of the great displacement from appropriate Bragg angle) and can be considered in the kinematical approximation. Qualitatively the phase sensitivity can be understood as the perturbation to the conventional kinematical diffraction induced by the spatial modulation of the wave field in the crystal due to dynamical diffraction - X-ray standing wave.

The approach discussed was used in the experimental studies of perfect crystals, crystal after ion implantation and III-V heteroepitaxial structures. Perfect crystals were used to exemplify the capabilities of the approach, for ion-implanted crystal we were concerned with the implantation-induced deformation of the subsurface layer [2], whereas in III-V structures we observed specific film-induced effect. Both synchrotron radiation and X-ray from conventional tubes were used in this studies. Various approaches to the two-dimensional collimation of the incident beam are discussed.

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HIGH ENERGY X-RAY MICROSCOPY TECHNIQUES WITH COHERENT SYNCHROTRON RADIATION

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Spatial coherence of the x-ray beam is essential for high resolution x-ray diagnostics particularly where microimaging and microfocusing techniques are concerned. Coherent and very intense x-ray beams provided by third generation of the synchrotron radiation sources such as the ESRF will bring a great impact on high energy x-ray microscopy, microholography and microinterferometry.

The high spatial coherence makes possible to realize holographic and phase contrast imaging in a simple transmission geometry identical to Gabor in-line holography setup. The phase-contrast images of different low density organic and non-organic materials were recorded with high contrast in the energy range 10-50 keV. It turned out that transparent for x-rays microobjects like fibers, particles and microcrystals can be easily visible. Considering the coherent properties of the beam evident benefits might be achieved in the development phase contrast tomography.

On the other hand, high spatial coherence of the beam imposes special requirements to all elements to be installed in the optical path such as crystals, mirrors, multilayers, filters and beryllium windows. A straightforward interferometry technique was applied to characterize the coherence of the x-ray beam and to study a coherence preservation by crystals, mirrors and multilayers.

The high coherence of the x-ray beam has to be taken into consideration for diffraction topography when the detector or film is placed at some distance from the sample. Moreover it was shown that invoking the phase contrast can provide an additional information about imperfections of the internal structure as well as about the topology of the sample surface.

The strong collimation of the ESRF beam provides an enormous advantage in efficiency over second generation sources in microfocusing optics like Bragg-Fresnel optics. The value of the transverse coherence of about 50 μm at 12 keV is comparable with the aperture of Bragg-Fresnel lens that allow to obtain effective focusing in a sub-μm range. Excellent compatibility Bragg-Fresnel optics with ESRF sources was clearly shown. Monochromatic focal spots of submicrometer sizes in photon energy range 6 - 100 keV were demonstrated. Microprobe and microimaging techniques based on Bragg-Fresnel optics were realized.

OBSERVATION AND TOMOGRAPHY OF PHASE OBJECTS WITH HARD SYNCHROTRON RADIATION X-RAYS

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The comparatively high spatial coherence of the photon beams from a third-generation synchrotron radiation source such as the ESRF, with very small electron beam cross-section, was recently shown to make the imaging of phase objects through Fresnel diffraction straightforward, and sometimes even undesirable [1,2]. Physically, the principle is identical to the defocusing mode of electron microscopy and to in-line Gabor holography in classical optics.

Unwanted contrast, due e.g. to thickness inhomogeneities in beryllium windows located several meters upstream of the specimen, appears on X-ray topographs. A random phase screen technique has been implemented to eliminate these spurious images, at the expense of slightly increased beam divergence, hence of a slight loss in image resolution. This approach is likely to be very useful in tailoring the beam for synchrotron radiation topography of not exceedingly perfect crystals at the topography beamline of the ESRF [3].

The useful applications are related with the possibility both of imaging light objects involving negligible absorption of hard X-rays but appreciable variations in optical path length due to thickness or compositional variations, and of obtaining additional information from absorbing samples. The experimental setup only involves, apart from the beam from the very sophisticated source, a perfect crystal monochromator, preferably in symmetrical Bragg setting, and a film placed at a distance of the order of 0.5 m from the specimen.

Novel possibilities arise in the extension of this phase imaging mechanism to tomography. This involves the use of a camera with good spatial resolution as detector, and a mechanical setup for specimen rotation. The data processing consists of two steps: first the contrast is converted to a two-dimensional map of the optical path-length variation [4,5], and then the three-dimensional distribution is reconstructed. In another configuration which is less demanding on the resolution of the camera, only the edges of the object show contrast. In this case the borders of the object can be reconstructed from the tomographic data [6] and the difficult inversion step to obtain the optical path length variation can be avoided.

Results obtained on test specimens and applications to materials science both in the stationary sample mode and in phase tomography will be discussed.

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MONOLITHIC DEVICES FOR HIGH RESOLUTION X-RAY DIFFRACTOMETRY AND TOPOGRAPHY

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Monolithic devices in X-ray diffraction optics are attractive because of their compactness, miniaturisation, and stability. Their concept was introduced more than two decades ago in connection with monolithic monochrocollimators [1,2]. In order to optimise the transparency of the system, one part of the crystal block was tilted relative to another. Lattice parameter alteration of the crystal block used to cut the monochrocollimator and the effect of refraction allow in a limited range such optimisation, too[3].

The aim of the paper is to present our recent results on two kinds of monolithic devices: (-n,n,m,-m) monochrocollimator for $CoK\alpha_1$ radiation, mainly its application in a simple multiple beam diffractometer, and X-ray magnifiers based on non-coplanar successive asymmetric diffractions, namely one with two successive diffractions for $CuK\alpha_1$ and the other for $CrK\alpha_1$ radiation and with three successive diffractions.

In the case of X-ray magnifiers, a comparison of the images obtained by standard X-ray tube and by synchrotron radiation will be given and much higher resolution obtained with synchrotron radiation will be discussed.

Based on the monochromator for $CoK\alpha_1$ radiation, a simple multiple beam diffractometer utilising simultaneous (004) and (115) diffractions has been devised and used to characterise InGaP/GaAs heterostructures. In comparison with standard high resolution diffractometry, substrate lattice parameter and tilt angle between substrate and epilayer can be obtained more easily.

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IMAGING OF BIOLOGICAL OBJECTS IN THE PLANE WAVE DIFFRACTION SCHEME.

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Some X-ray techniques utilizing a refraction on boundaries between media with different densities are used now for testing an inner structure of noncrystalline objects. A sufficiently detailed description of them one can find in [1]. All methods are based on an extreme sensitivity of multicrystal scheme of x-ray diffraction to small deviations of the beam due to refraction in an object having some structure elements which was placed between crystals.

The Phase Dispersion Imaging technique presented here is characterized by using a pseudoplane primary beam with divergence of about 0.1″ for object illumination and a crystal analyzer installed in Laue position behind the object. We have no doubts that Laue position of analyzer is more sensitive to small density gradients than Bragg one [2]. Owing to this we observed boundaries separated media with density variations of about 1% - 3% and soft tissues in biological objects [1, 3]. When going to $AgK_{\alpha 1}$ radiation and large crystals (monochromators and analyzer) we got possibility to examine laboratory animals: white mice and rats.

In spite of insufficient source power (1,5 kW) we tested not only cadavers but also alive animals put asleep. Costal cartilage, meatal and tubal cartilage are revealed on number of images together with inner organs: lung, liver stomach, etc. It can be reasonably supposed that lymphatic ducts are also visible on PDI images with high contrast. The most significant result is that in some experimental conditions we registered vessel system - venous and arterial. The last advantage allows to predict that PDI technique has a very high potential for number of diagnostics.

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AN ADVANCED X-RAY HRD STUDY OF MICRODEFECTS IN Si DOPED GaAs BULK CRYSTAL

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The GaAs single crystal is a very perspective material due to wide optoelectronic applications. Unfortunately a large density of microdefects (MD's) has been observed in nearly dislocation-free single crystals [1]. Since microdefects within the GaAs substrates can act as defect sourses in the epitaxial layers, experimental methods capable of revealing MD's in GaAs single crystal are of great interest.
The X-ray diffuse scattering is a powerful method for the investigation of the defect structure, size, concentration and type [2]. But it is well developed for one microdefect type crystal. When there are different microdefects in a bulk the XRD is not practised on a large scale because results interpretation hardness. The problem can be advanced by studying of diffuse scattering in the asymptotic region. There is possibility to distinquish diffuse scattering from interstitial and vacancy microdefects in different angular parts.
We will report our experimental XRHRD investigations of MD's in Si doped (up to concentrations $c \sim 1.6\times10^{19}$ and 4.5×10^{19} cm $^{-3}$) GaAs single crystals grown by HBG method. The experiments have been performed with a full automatic triple-crystal diffractometer using hard and software produced by MATEX Ltd. Both coherent and diffuse parts of X-ray total diffraction scattering were used for MD's characterization near (004) symmetrical Bragg reflection on copper radiation. Theoretical simulation and treatment of experimental resuts were done accordingly.
It was found for the both samples what the main MD's type is interstitial dislocation loops with Burger vector (1/2)[110] what are distrubuted in (110) and (111) planes. For every sample the loops are devided for two groops with average radii 27 nm, 8 nm and 53 nm, 20 nm for c(Si)=1.6×10^{19} and 4.5×10^{19} cm $^{-3}$ respectively. The static Debye-Waller factor of a average lattice was used to obtain the main defect concentrations.

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Influence of Point Defects and Dopants on the Lattice Constant of GaAs, AlGaAs and GaN

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The lattice constant is one of the most basic parameters characterising every material. It can be measured with a very high accuracy (1-10 parts per million) by using the X-ray diffraction methods. For semiconductors, the lattice constant depends on stoichiometry, dopant concentration, as well as on the free-charge concentration (via the deformation potential). The main goal of our research was to separate these factors in GaAs:Si, Te, AlGaAs:Te and GaN possessing a high concentration of native defects (presumably, nitrogen vacancies).

In our experiments we performed measurements at variable temperature and illumination. For AlGaAs layers doped with Si, Te we could change the free-electron concentration by white-light illumination and emptying deep DX centers (at 77 K). However, this phenomenon is accompanied by a rearrangement of atoms forming this defect and an additional lattice relaxation. The magnitude of this relaxation could be evaluate by a comparison with EL2 defects in GaAs, for which no change of the free-electron concentration occurs. As a result, we could extract an information how free-electrons change the lattice constant and verify the values of the energy-gap deformation potentials.

The other important result from these measurements was an evaluation of changes of the thermal expansion caused by doping. The changes are significant and should be always taken into account when the lattice constants at 295 K are considered.

The other examined material was a wide-band-gap semiconductor, gallium nitride. The measurements were performed on the unique bulk samples (grown at high pressure of about 15 kbar) of different free-electron concentration at variable temperature.

DAFS-experiments at GaInP/GaAs-layers using superlattice reflections

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Ordering during epitaxial growing has been reported for many III/V alloy systems. It is interesting because of its influence on the performance of electronic devices developed on base of these materials. For $Ga_{0.5}In_{0.5}P$ epitaxial layers at appropriate conditions during growing on (001) GaAs substrates on two of the four possible {111} planes a CuPt-like structure of the column III atoms has been reported [1].

The idea of our experiments was to investigate the structure of the ordered regions separately. **The new aspect was to use anomalous scattering of the superlattice reflections within an energy range including the Ga-K-edge.** This corresponds to DAFS-experiments (Diffraction Anomalous Fine Structure) [2]. The smooth intensity run gives a certain confirmation of the crystallographic structure and the fine structure contains information especially of the neighbourhood of the Ga atoms in the ordered regions. The most important aspect is, that **one can get these information realy from the ordered regions (less then a percent of the whole material) selected by their (superlattice) reflections.**

The experiments we carried out at the RISØ-goniometer at station BW1 of DORIS III at HASYLAB using a Si (111) double crystal monochromator and self prepared peltier cooled Si-pin photodiodes as detectors for scattered and fluorescence radiation.

Figure 1 shows on the right the corrected run of the scattered $Ga_{0.5}In_{0.5}P$ superlattice reflection intensity in the vicinity of the Ga-K edge (about 10370 eV) and on the left the fluorescence intensity run (XAFS) simultaneously measured on top of the sample. It is used for the correction of absorption caused by the interaction of the radiation with the whole material on the path to and from the ordered regions.

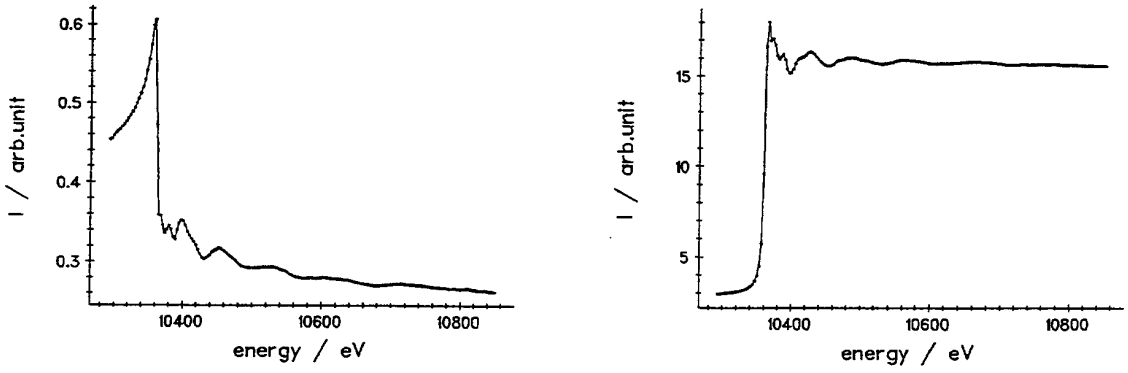

Fig.1: Scattered $Ga_{0.5}In_{0.5}P$ superlattice reflection intensity (left) and fluorescence intensity (right)

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A NEW STATISTICAL METHOD FOR THE SIMULATION OF DOUBLE CRYSTAL X-RAY ROCKING CURVES

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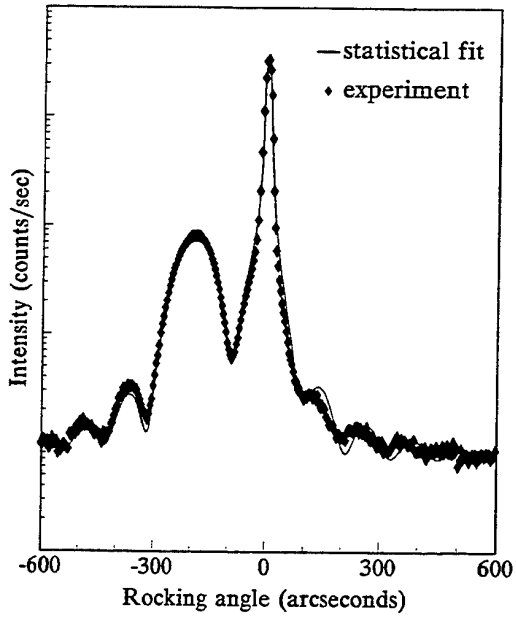
Quantitative analyses of double crystal x-ray rocking curves typically involve comparisons between an experimental data set with profiles calculated from either dynamical or kinematical diffraction models. A difficulty with this approach is that the comparison is usually performed by visual inspection, with the input to the model adjusted according to the experience of the individual performing the analysis and the "quality" of the fit between model and experiment based on a subjective assessment. We have developed a statistical method for the optimization and analysis of rocking curve simulations for epitaxial thin films in the Bragg case which circumvents this problem. In this approach (written for the MATHEMATICA® programming language system) the diffracting medium is modeled as a set of lamellae on a semi-infinite substrate, each with a unique x-ray susceptibility, following the method of Bartels. After inputting an initial user-specified structure, the structure parameters (structure factor, layer thickness, *etc.*) are converted into dynamical diffraction variables $C=\frac{(x-\eta)\cos k\overline{\eta}-i\overline{\eta}\sin k\overline{\eta}}{\overline{\eta}\cos k\overline{\eta}-i(x-\eta)\sin k\overline{\eta}}$ and $G=\eta+C\sqrt{\eta^2-1}$, where η is the deviation parameter from dynamical diffraction theory, $\overline{\eta}=\sqrt{\eta^2-1}$, $k=(r_e\lambda^2/\pi V_c)(FF^*)^{1/2}$, and all other terms having their usual definitions. It is these parameters, rather than the usual quantities (composition, thickness, *etc.*), that are used to fit the experimental intensity by calculating $I_{fit}=GG^*$. The fit is then optimized by a gradient-descent on the error surface to minimize the difference between the calculated and observed intensities. Allowed values are constrained by the requirement of a unique Bragg angle for the layer and a Darwin-Prinz relationship between the real and imaginary components of the deviation parameter. A confidence limit obtained from the experimental noise level is used as a criterion for statistical consistency of the fit with the data. The result is a statistically-significant range of structural parameters which fall within the defined noise envelope of the experiment. This method has the advantage that fits are not limited by specific assumptions about the layer structure or electron density, while the use of transform parameters that are directly utilized by the dynamical simulation (G,C,η,k) increases the robustness of the fit. Statistically-significant fits to a variety of experimental rocking curves (such as the 004 rocking curve from a 0.16 μm Ge layer on GaAs shown here) demonstrate that good agreement with experimental data can be achieved.



EFFECT OF UNIAXIAL STRESS ON THE LATTICE SPACING OF SILICON AT LOW TEMPERATURES

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Recent measurements by high-angle double-crystal X-ray diffractometry (HADOX) in a temperature range 100-300 K showed that the lattice spacing of silicon is stress sensitive. In addition, the lattice spacing measured by the Bond method showed anomalous temperature dependence below 100 K even without applying external stresses. The purpose of the present investigation is to examine the effect of uniaxial stresses on the lattice spacing of silicon at low temperatures, and to obtain information about the stability of the crystal lattice. The experiment was carried out with a HADOX diffractometer operated in the 2θ-resolved mode. By this method we can determine the change in the lattice spacing and the change in the orientation of the scattering vector independently. Specimens, prepared from a dislocation-free non-doped FZ crystal, were in such a form that prevent the stress at one face, glued to the cryostat, from propagating to the spot exposed to X-rays. Measurements of the 444 diffraction with CuKα_1 radiation at $\theta \sim 79°$ were made in a temperature range 40-300 K; the uniaxial stress was applied in the direction perpendicular to the scattering vector in [111], namely parallel to either [$1\bar{1}0$] or [$11\bar{2}$], in a range up to 5.4 kPa. The incident beams were monochromatized with 444 of another crystal of silicon. The results show that the temperature dependence of the lattice spacing d(T), and of the full-width at half-maximum (FWHM) of the rocking curve of the diffraction w(T), depends on the direction of the external stress. The most striking effect is discontinuous changes in w(T) observed at 235 and 140 K with the stress applied along [$1\bar{1}0$]; the w values between the two temperatures are about two times as large as those at other temperatures. The change at 235 K is consistent with previous observations. For the stress along [$11\bar{2}$], w is constant at the theoretical value for a perfect crystal of silicon. In both cases, d(T) curves depend on the stress, and more sensitive when the stress is parallel to [$1\bar{1}0$]. From these results, it is concluded that the lattice spacing of silicon is stress sensitive, in particular at low temperatures, and that the effect of stress is anisotropic. The relative change in d values $\Delta d/d$ for the stress of 5 kPa is of the order of 10^{-5}.

Investigation of X-ray interference fringes observed on SIMOX structures

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X-ray interference fringes can for example be observed on bicrystals with an implanted layer as spacer. They may originate from a lattice parameter difference ("Moiré fringes") or from a rigid body translation ("translation fault fringes") between substrate and layer. Topographs of samples produced by SIMOX processes (Separation by IMplantation of OXygen) [1] exhibit pronounced fringe systems. Both their geometry and the intensity profiles recorded on the topographs were analysed in the present study.

In transmission geometry the use of white synchrotron radiation allowed to collect topographs from more than ten symmetrical and asymmetrical reflections in a single exposure. These fringes can be interpreted as Moiré fringes [2]. From their spacings, measured on topographs of different reflections from one sample, the tensor of the relative deformation between layer and substrate is determined. This relative deformation ranges from less than 10^{-9} to some 10^{-7}, depending on the SIMOX production process.

In reflection geometry, using a rotating anode and a curvable collimator crystal to adjust for sample bending [3], the whole sample could be imaged for several reflections. This setting is very sensitive to thickness variations in the Angstrom range in the spacer layer.

Finally, the interaction between interference fringe and defect contrast is studied.

Thanks to the consequent use of white synchrotron radiation, a curved collimator technique and a consistent interpretation procedure for the topographic results, in the present work more experimental parameters could be determined with good precision than in any previous X-ray Moiré or translation fault fringe analysis.

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X-ray Diffraction Study of Porous Silicon Layers Etched on (111) Oriented p^+ Substrate

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The porous (PS) silicon layers were prepared by a standard anodic dissolution process of silicon in HF solution on (111) oriented substrates. Initial low resistivity silicon wafer was covered with 20 μm thick boron-doped CVD layer (resistivity ~ 10 Ωcm) and was used as actual substrate for PS etching. Range of samples were examined with different maximum of photoluminescence signal ranging from 500 nm to above 800 nm. High resolution X-ray diffraction in triple axis arrangement, SEM, TEM and FTIR experimental methods were used to correlate structural and luminescence properties of such PS layers. Apparent lattice parameter difference correlated with porosity and maximum luminescence signal strongly supports quantum confinement model of the photoluminescence phenomena in PS. The role of oxygen in PS [1,2,3] photoluminescence was also further studied.

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STUDY OF X-RAY SCATTERING FROM MICRODEFECTS IN BULK AND THIN LAYER MATERIALS.

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Knowledge of structure perfection of as-grown single crystals or those after processing, such as ion-implantation, used for fabrication of devices in semiconductor technology, is required for better control of electronic properties. Microdefects, point defect clusters or dislocation loops, are common in these materials. X-ray scattering measured from these materials can be analysed to provide quantitative description regarding their nature, size and concentration. High resolution triple-axis technique was employed to measure anomalously transmitted X-ray scattering (in triple crystal configuration) from InP wafers and Bragg reflected scattering was measured in seven crystal configuration from thin layer formed in InSb after implantation with In ions. The analysis of the X-ray scattering is based on the calculation of the displacement field from the defect [1] and subsequent simulation of the diffuse scattering [2] for comparison with the experimental results. In the case of bulk InP crystal two dislocation loop systems {111}<110> and {110}<110> were treated. Considering uniform distribution of dislocation loops on {111} and {110} planes with all possible Burgers vectors directions <110>, the simulated symmetric and asymmetric parts of scattering showed better agreement with the experiment for interstitial loops of 2 μm diameter belonging to {111}<110> system.

In the case of as-implanted InSb crystal it was found that the measured rocking curves can not be explained only by strain depth distribution described by asymmetrical Gaussian model and the static Debye-Waller factor accounting for lattice disorder and having the same depth dependence behaviour. Additional scattering calculated from dislocation loops belonging to the system (111)[111] provided better agreement with experiment and increase in the size of these defects could explain the broadening around the Bragg peaks observed from the InSb sample implanted in the presence of the permanent magnetic field.

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Characterization of microdefects in GaAs crystals with high resolution X-ray diffractometry

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The aim of the present work was to correlate the electric characteristics of Te doped gallium arsenide ($1\cdot10^{17}\ cm^{-3} \le n \le 1\cdot10^{19}\ cm^{-3}$), grown by LEC technique, with the defect contents of the samples. We also performed measurements for some Si, Ge and Ge+Te doped samples as well as undoped ones, grown by various methods (HB, VGF and LEC). In order to characterize the defects High Resolution X-Ray Diffractometry [1] was used. Measurements of diffuse scattering intensity maps around the reciprocal lattice points and plane wave reflection topography were employed [1,2]. For most cases in the range of $1\cdot10^{17}\ cm^{-3} \le n \le 6\cdot10^{18}\ cm^{-3}$ the isointensity contours in the maps exhibit strong resemblance to each other as to their general character. However, a close analysis of the slopes in the log-log dependences of the intensities on the reciprocal lattice vector $\Delta q_{\|}$ reveals distinct differences for the various cases studied. For free electron concentrations close to $1\cdot10^{19}\ cm^{-3}$ the character and symmetry in the isocontours are changed from the elongated images perpendicular to $\Delta q_{\|}$ to contours which show fourfold symmetry. An analysis of these maps using simulations [3-6] based on the kinematical theory of X-ray diffraction will be presented. Although the precise determination of the defect type is rather complicated, we can conclude that formation of extended-type defects does influence the relation between the concentration of the dopant atoms and the free electron concentration.

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POSTER SESSION

1. Advanced X-ray topography methods

2. Instrumental developments

3. Applications to crystal growth

4. Applications to technological processes

5. Advanced X-ray Diffraction methods

6.Theoretical models

7.Applications to surface and interfaces

8.Applications to physical phenomena

ABSTRACTS OF POSTER CONTRIBUTIONS

Dynamical Diffraction Effects in X-ray Topography.

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The X-ray topography is widely used in the study of the crystals. In some cases the quality of the crystals allows to found the dynamical diffraction effects. These effects give more detail information about structure of crystal. In this work present the realization and the employment the dynamical diffraction in the crystals HTSC , ZnS and $LiNbO_3$ for the study of their structure's by the X-ray topography methods.

The X-ray anomalous transmission effect (Borrmann effect) in Nd_2CuO_4, $(LaSr)_2CuO_4$ (Tc=15K), $EuBa_2Cu_3O_{7-x}$ and $YBa_2Cu_3O_{7-x}$ (Tc=92K) single crystals has been observed. With the help of X-ray topography we have shown that the grown in alundum container the $EuBa_2Cu_3O_{7-x}$ crystals are noncentrosymmetrical. They have the polarity axis along <100> and divided into antiphase domains. The result of existence of the X-ray anomalous transmission effect in strongly distorted crystals have been obtained on the $EuBa_2Cu_3O_{7-x}$ crystals. It shows that the theories of Borrmann effect demand the future development.

The X-ray technique has been used to investigate single ZnS crystals ($(F\bar{4}3m)$, a=5.408Å) grown according to Bridgman by vertical oriented crystallization under an argonpressure of 5-6 MPa. These crystals consist of microtwins misoriented to each other 60° about the axis <111>. Because for stacking faults, some reflections in single ZnS crystals are extended along <111> and many of them cross the Ewald Sphere always. The topography may be realize on these reflections. Some reflections show the multiple diffraction. This is manifested in extra narrow lines on the topographs and additional reflections on the diffraction patterns. We cannot elucidate these phenomena with traditional diffraction methods.

Also the topographs in white radiation and the angular scanning topographs of $LiNbO_3$ crystals has been analyzed. The obtained results on the $LiNbO_3$ crystals and ZnS crystals has been comparison. This comparison shows the different nature of the addition lines on the topographs of ZnS crystals and the lines on the topographs of the multiple diffraction in white radiation of $LiNbO_3$ crystals.

The model of the multiple diffraction based on the assumption that the multiple diffraction effect increases when a phase invariant equal's zero has been considered. This model describes the experiment results for ZnS crystals satisfactorily. The considered phenomenon should be characteristic for all layered crystals in which diffraction peaks are associated with the thickness of the layers or at plastic deformation, when several atomic layers shift simultaneously. Some of the possible objects for the realizes of this phenomenon are the defect HTSC crystals.

QUANTITATIVE ANALYSIS OF SCREW DISLOCATIONS IN 6H-SIC SINGLE CRYSTALS

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ABSTRACT

Screw dislocations along the [0001] axis in 6H-SiC single crystals have been studied extensively by Synchrotron white beam X-ray topography (SWBXT), Scanning electron microscopy (SEM), and Nomarski optical microscopy (NOM) [1]. Using SWBXT, the magnitude of Burgers vector has been estimated from the diameter of diffraction-contrast images of dislocations in back-reflection topographs, and confirmed in transmission [illegible] secondly from magnitude of the tilt of the lattice planes on both sides of dislocation core in both projection and section topographs. SEM results reveal that micropipes in the form of hollow tubes run through the crystal emerging as funnel-like features on the as-grown surface, with their diameters ranging from around 0.1 to a few micrometers. Correlation between topographic images and SEM micrographs shows that micropipes are screw dislocations with Burgers vector magnitudes typically from $2c$ to $7c$ (c is the lattice constant along [0001] axis). The Burgers vector magnitude of the screw dislocations, b, and the diameter of the associated micropipes, D, are fitted to Frank's prediction for hollow core screw dislocations [2]: $D = \mu b^2/4\pi^2\gamma$, where μ is shear modulus, γ is specific surface energy. Statistical analysis of the relationship between D and b^2 shows that it is approximately linear, basically confirming Frank's prediction, and the constant, γ/μ, can be obtained from the slope. The mean value of γ/μ ranges from 0.011 to 0.016 Å. Using such values, the criteria for dislocations to form hollow cores are discussed, and the extrapolation to elementary screw dislocations ($1c$) is presented and discussed.

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Simulation of Synchrotron White Beam Topographs. An Algorithm for Parallel processing: Application to the Study of Piezoelectric Devices.

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Recent experiments have shown that synchrotron white beam topographs may be simulated describing the incident beam as a distribution of incoherent point sources. This allows to use the theorem of reciprocity to simulate the images.

We present a new algorithm for the integration of Takagi-Taupin equations taking into account the fact that X-Ray diffraction is a parallel phenomenon. Maxwell equations show that the propagation of the waves is independent in each incidence plane. It is thus possible to compute in parallel the propagation of the waves in different planes. Two algorithms are presented: the first one for multiprocessors machines where the processors share a common memory, the second one for massively parallel computers. The numerical method as established by Carvalho & Epelboin (1993a) has been modified to be able to vectorize the computation and to make it as efficient as possible with modern superscalar and array processors. The simulation of the image of a defect has been divided into two independent parts. In the first one, one computes the derivatives of the deformation inside the crystal, in the second one these results are used to simulate the image. This allows to rapidly change the model for a defect which was not feasible in all previously written simulation programs since the computation of the deformation was part of the simulation.

The study of stroboscopic images of the propagation of acoustic waves in piezoelectric devices is given as an example of the possibilities of this new program. The devices are circular plates from a quartz AT-cut crystal. The experimental topographs have been recorded by B. Capelle, Détaint & Zarka (1995). The models describing the deformation inside the crystal is made of a development in Bessel functions (Détaint, Carru, Schwartzel, Joly, Capelle, Zarka, Zheng & Philippot, 1992). The agreement between the experiments and the simulation is good except in the center of the resonator, which may be explained by the imperfections of the excitation electrodes.

SOFT X-RAY TOMOGRAPHY ON IR-T1 TOKAMAK

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ABSTRACT

A soft x-ray tomography diagnostic has been built and operated on the IR-T1 Tokamak with the use of an 23-channel array of detectors. Cormack's method is used for reconstructing the images of soft x-ray emissivity distribution in the poloidal cross-section of IR-T1 Tokamak. The detector system consists of one horizontal array of silicon surface barrier diodes on a circular arc view IR-T1 Tokamak plasmas through a thin beryllium window (in a fashion similar to that of a pin-hole camera) , to provide poloidal imaging of soft x-ray emission in the 900 ev- 20 kev range. Each detector has an active area 3×20 mm^2. The radial resolution is 1 cm and the detectable space range in radial direction of plasma cross-section is approximately 0.9 , which is normalized to the limiter radius. The signals are digitized every 4μs or 8μs by use of 8 bit transient recorders and the frequency response of detector-preamplifier system is from 10 Hz to 250 kHz.

This method has been used to study the internal disruptions and large m = 1 oscillation. Since reconstructing the plasma image containing the m > 0 perturbation modes the chord-integrated signals sampled in different poloidal direction at every certain radial position are required, therefore , it is necessary to assume that plasma column rotates rigidly in poloidal direction under the condition of one detector array, so as the successive profiles from one detector array can equal to the profiles sampled simultaneously in different poloidal direction. The rotating frequency of plasma column is determined from the frequency of m = 1 oscillation on the soft x-ray signal. On the IR-T1, the m = 1 frequency is about 19 kHz - 25 kHz according to the tokamak discharge parameters. The reconstructed images have shown that the large m = 1 oscillation is caused by the rotation of plasma column.

TOPOGRAPHICALLY-SENSITIVE LATTICE PARAMETER MEASUREMENTS ON DIAMONDS

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This talk has three aims. The first is to itemize the major known impurities in natural and synthetic diamond and to indicate their importance in modifying the unique mechanical, optical and thermal properties of diamond. The second aim is to offer a brief critical review of world-wide efforts to determine the effect of impurities on the lattice parameter of diamond. The third aim, and principal feature of this contribution, is to report on recent (and largely unpublished) work done in UK on this topic.

Regarding impurities, the best-known impurity in both natural and synthetic diamond is nitrogen, which can occur in several states. These range from atomic dispersion, singly-substituting for carbon atoms in the diamond matrix (which is the manner in which nitrogen principally exists in synthetic diamond manufactured by the conventional high-pressure, high-temperature technique), through various 'small cluster' states, to incorporation (albeit as a minor constituent, according to recent studies) within those planar defects, the well-known 'platelets' lying on diamond matrix cube planes, that give rise to the anomalous 'spike' diffuse X-ray reflexions exhibited by most natural diamonds. For some nitrogen aggregation states plausibly-dimensioned structural models have recently been computed. Moreover, on the microscopic scale, transmission electron microscopic work in progress at Bristol (employing the Large Angle Convergent Beam Diffraction technique) is providing more exact figures for the diamond matrix displacement produced by **individual** platelets. To assess the microscopic models, precise X-ray measurements of the macroscopic lattice parameter are needed. However, such measurements are commonly bedevilled by the inhomogeneity of diamonds, manifested in impurity zoning, and by strong growth-sectorial dependence of impurity concentrations. Therefore it cannot be too strongly emphasized that for measurements to be scientifically meaningful they must be performed with **topographic sensitivity**, working on crystal volumes fully characterised by X-ray and cathodoluminescence topography and by infrared absorption microscopy. Examples of recent work fulfilling these conditions will be presented. One set involves a modern version of Lonsdale's pseudo-Kossel, divergent X-ray beam method, now using X-ray excitation by electron probe in the SEM; other experiments involve double-crystal topography with synchrotron radiation.

EFFECT OF THE ANNEALING CONDITIONS ON THE STRUCTURE OF $Pb(Zr_XTi_{1-X})O_3$ THIN FILMS DEPOSITED BY SOL-GEL

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Ferroelectric thin films such as lead zirconate titanate (PZT) are of considerable interest for the realization of memory devices such as nonvolatile ferroelectric random access memories (FERAMs) since they display a spontaneous electrical polarization, that can be switched on between two stable states, and a high resistivity and radiation hardness. In these devices the ferroelectric thin film is integrated as a ferroelectric capacitor together with semiconductors devices. In the present work we report preliminary results on the effects of annealing conditions on the structure of $Pb(Zr_XTi_{1-X})O_3$ thin films deposited by the sol-gel method in order to investigate the formation and the evolution of the ferroelectric perovskite phase. PZT films with x= 0.52 were deposited on Pt/TiO_2 substrate using multilayer spin coating. Each PZT layer was heat treated at 300°C in air to pyrolyse the organic constituents of the gel. After deposition films were annealed at temperatures ranging from 500°C to 700°C for 60 minutes in O_2. Finally, a gold top electrode was evaporated and defined by classic photolithographic process to obtain the ideal capacitor structure. Samples were characterized by scanning (SEM) and transmission (TEM) electron microscopy techniques and by grazing incidence X-ray diffraction (GID) performed at different angles of incidence of the X-ray beam with respect to the sample surface in order to study the depth profile of the films. Results showed that at 500°C the sample is constituted of a mixture of the pyrochlore type $Pb_2Ti_2O_6$ non-ferroelectric phase and the perovskite type $Pb(Zr_{0.52}Ti_{0.48})O_3$ phase with a prevalence of the pyrochlore one. On the contrary, at 550°C the sample is completely constituted of the perovskite phase without variation of the stoichiometry in the depth of the film. At higher temperatures samples are still constituted of the perovskite phase which does not present any preferential orientation. It is worthnoting that the transition temperature (550°C) to the ferroelectric phase exhibited by our samples is relatively low if compered to the data reported in literature. In conclusion, it was demonstrated that it is possible to obtain PZT thin films by the sol-gel method exhibiting good ferroelectric properties at relatively low temperatures.

X-RAY DIFFRACTION TOPOGRAPHY AND LATERALLY RESOLVED DIFFRACTOMETRY WITH CCD REGISTRATION

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X-ray diffractometry and topography are well–established tools for investigation of real structure of single crystals. However, a constantly growing complexity of objects under investigation requires new development of methods and technical means of their realization.

In this work a two–dimensional CCD detector was used for registration in synchrotron radiation topography in symmetrical as well as grazing–incidence geometry and multiple–crystal diffractometry. The experiments were performed at the beamlines ROEMO I and CEMO of HASYLAB, using double-crystal Ge / Si monochromators. A CCD detector ("Hamamatsu Photonics") with a 1000×1000 array, a pixel size of $12 \times 12 \mu m$ and direct registration of radiation was used.

In X-ray topography, long–range images from a III/V heteroepitaxial films as well as lateral structures on InP and $LiNbO_3$ were investigated. The formation of contrast from lattice strains in GID topography around the critical angle of total external reflection was also studied.

Simultaneously with topographical measurements, sequences of images were taken with a small step at different deviations from the Bragg angle. The CCD detector provided a unique opportunity to monitor the diffracted beam and choose places of interest on the sample surface. This allowed, due to a good lateral resolution of the detector, to reconstruct the reflection curves in different points for the samples, which could be otherwise investigated only with a complicated microbeam technique.

It is shown, that modern CCD devices combine a good lateral resolution and large dynamical range with a large–area of registration and an excellent sensitivity to X-rays at wavelengths $\sim 1\mathring{A}$. They enable one to investigate interactively complicated structures on single crystal wafers and to make diffractometric measurements in the regions of interest with a lateral resolution $\sim 20 \mu m$.

It is shown, that by variation of incidence angle in GID X-ray topography one can separate the refraction effects and provide for layered structures a lattice parameter resolution better than $\Delta d/d \sim 10^{-3}$ at information depths down to tens of nanometers.

SYNCHROTRON RADIATION TOPOGRAPHY OF SILICON ON INSULATOR MATERIALS

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Separation by IMplantation of OXygen (SIMOX) and wafer bonding are the most important of the existing techniques to produce Silicon On Insulator (SOI) structures [1]. Despite technological improvements, defects remain present in the material. In the upper silicon layer, these defects are crystalline defects such as dislocations, but also long range strain fields may appear. These defect structures can be studied non-destructively by X-ray topographic techniques [2]. Such methods are especially well adapted to study SOI materials with dislocation densities less than 10^5 cm^{-2} (the practical detection limit of TEM). With these low dislocation densities, reaching even values of 10^2 - 10^3 cm^{-2} in some production processes, topography provides more information (e.g., Burgers vector) than the usual selective etching technique which, in addition, is destructive.

The defect structure of SOI-samples was studied at the ESRF with two methods: white beam and double crystal X-ray topography. The samples investigated were produced by the wafer bonding method and by standard, multi-implantation, and low dose SIMOX processes followed by different heat treatment procedures.

Threading dislocations in the upper silicon layer of SIMOX samples were observed although the epitaxially grown upper layer is only a few μm thick (about ten times less than the Pendellösung or extinction distance). Often the dislocations form pairs (separated by a few μm) with antiparallel and orthogonal Burgers vectors. Their density and contrast were investigated as a function of several experimental and process parameters. One surprising result is that the width of the dislocation image does not depend on the diffracted wavelength. Another interesting feature is the interaction of the defect contrast with that of Moiré fringes. Both these results support the idea that the contrast is mostly dynamical, even though $\mu t << 1$.

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TOPOGRAPHY IMAGES OF DEFECTS IN ACOUSTICALLY EXCITED CRYSTALS.

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In the given research the mechanisms and regularities of X-Ray diffraction image formation of a different type defects in an acoustically excited crystal depending on the amplitude W and ultrasonic wavelength λ_s are considered. Simulations of the ultrasonic transversal wave parameters influence on formation of the diffraction images of dislocations, microdefects and different type of combined distortions of a crystal lattice in section and plane-wave topographs are performed by solving numerically the Takagi equations. At excitation in crystals of ultrasound with a wavelength, equals to the extinction length (Λ), a significant transformation of diffraction contrast of such images occurs. The deformation depth oscillations creation under the resonance condition ($\lambda_s = \Lambda$) increases the topography sensitivity to separate defects. Details of X-Ray acoustic interaction are defined by the ratio λ_s / Λ , as well as by W value. The change of amplitude in the case of $\lambda_s = \Lambda$ is caused by periodic passing of depth oscillations minima of a diffracted wave coherent part through a defects position. That provides the increase of the defects image sizes both for kinematical and dynamical parts of scattering [1,2]. This effect may be applied for revealing of such defects in rather thick crystals.

In the case of $\lambda_s = \Lambda$ the amplitude dependences of integrated intensity I (W) and their space distributions I (x) are experimentally investigated in crystals, which contain swirl - defects. Extinction length, static Debye-Valler factor L, coefficient of diffusion absorption and level of deformation of a crystal lattice are determined. The characteristic sizes of microdefects and their density per unit of volume are found.

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NEW FEATURES OF TOPOGRAPHY WITH HIGHLY COHERENT X-RAY BEAM.

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The traditional X-ray diffraction topography as a method of investigation crystal lattice perfection by means of obtaining photo images still withstand the tendency of replacing it with the modern computerized techniques and sophisticated detector systems because of its simplicity and very high resolution. With appearance of new generations of synchrotron radiation sources the topography and tomography becomes more and more important as the nondestructive methods of studying massive balks of matter. With the advent of third generation synchrotrons this techniques have got new features connected to the advanced properties of the source. The traditional topography never considered the characteristics of the incident radiation in terms of coherency except of monochromatization degree. Nevertheless the experiments investigated the coherent properties of the x-rays were performed by means of topography, for instance propagation and diffraction of the spherical wave [1,2] or influence of the optical setup on the Pendellösung effect [3,4]. As far as X-rays are electro-magnetic waves the notations and terms of the visible light optics are acceptable. But due to specificity of the diffraction and refraction phenomena for X-rays they sounds different.

The modern synchrotron radiation source, like ESRF, has features never observed before, namely very small source size, highly collimated beam and a large distance to sample. These allows one first to vary the sample-to-film distance still keeping the resolution as high as about 1μm [5], and second an additional information may be obtained taking into account the coherent properties of the beam. The aim of the present work is to illustrate on a qualitative basis how the topography image can be transformed due to the interference phenomenon in air after a sample.

The experiments were conducted at the ESRF Optics beamline BL10/D5. The BL10 is a general purpose beamline on a bending magnet with a high resolution triple crystal diffractometer situated about 40m downstream the beam. The source size is about 120μm by 240μm in vertical and horizontal directions respectively. Distance between sample and photo film can be varied up to few meters. The samples under investigation were varied from the crystals with strongly deformed areas, namely InP/InGaA structures grown by selective area epitaxy in-between oxide strips, up to almost perfect substrates with very weak and localized deformation fields in the near surface layer caused by thin oxide film (SiO_2 on Si). The topographs were obtained using 12keV radiation and either single or double crystal layout. For example, SiO_2 film on Si substrate generates stresses localized in a narrow region around the oxide edge with typical value $\Delta d/d=10^{-7}$-10^{-8} for standard 0.6μm thickness. This value is already close to the resolution limit of the plane wave topography. Invisible at the observation plane close to the sample this defect becomes very pronounced at the distance 0.5m. Generally speaking the sensitivity of the topography method to detect the weak and localized deformations inside a perfect substrate can be improved by varying the distance sample-to-detector. The second point is that the phase information lost in the vicinity of sample position can be visualized at certain distances which is in fact analogous to the recording of in-line holographic image.

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SINHROTRON DIFFRACTION TOPOGRAPHY POSSIBILITY FOR OBSERVATION OF SEMICONDUCTOR SUPERLATTICE PERIOD INHOMOGENEITY

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The development of the Synhrotron diffraction topography method previously reported in [1,2] of investigation of superlattice (SL) structure parameter inhomogeneity across the surface of the wafer is presented. With the satellite number increasing, the diffraction contour behaviour becomes to be mainly determined by distortions of the SL period and permits to indicate the gradient of their distribution. The Synhrotron diffraction topography method involves two stages [1]. The first one is the qualitatively observation of the SL structure, and the second one, which is more time consuming, is the calculation procedure. The proposed variant of the SL period inhomogeneity determination can be used at the first stage and gives us the possibility of quickly observation of the mostly distorted areas in the SL structure.

For the investigation, the topographs obtained using the satellites with positive and negative numbers are needed. Experimental results has been obtained using SL of AlAs/AlGaAs on the (001) GaAs substrate. The equipment of Siberian Synhrotron Radiation Center was employed. The topographs using the satellites with numbers of -2 and +3 were registered. For the crystal position at which the projection of the incident and the reflected beams at the crystal surface is parallel to the period inhomogeneity gradient, the behaviour of the diffraction contours with the sample rotation around the Bragg axis is similar for the both satellites. But for the crystal position at which this projection is perpendicular to the gradient, the diffraction contours move in the opposite directions. The movement of diffraction contours demonstrating this effect as well as the behaviour of diffraction contours for other satellites was recorded by video and can be shown at the Symposium.

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X-RAY TOPOGRAPHIC ANALYSIS OF THE VICINAL SECTORIALITY IN KDP GROUP CRYSTALS

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The vicinal sectoriality (VS) is a special kind of three-dimensional impurity inhomogeneity in crystals especially grown with high rates. This is shown to be the primary source of the optical transmitted-wavefront distortions. Information about the elastic state of the vicinal sectors and boundaries is important for understanding the mechanisms of inhomogeneities formation and for improvement of the crystal quality. The plane wave X-ray topography technique has been used for analysis of VS in relation with growth parameters of KDP crystals.

Primary studies showed that one vicinal sector of three-angle vicinal pyramid on {101} faces differs significantly from two others and its deformation state is described not only by lattice parameter change but a small rotation also. Quantitative measurements of these values were carried out by means of computer image processing system with use of two pairs of symmetrical Bragg (reflection) topographs (404 reflection, $CuK\alpha_1$). The following data were obtained for crystal grown with rate of 15 mm/day: $\varepsilon_{zz}= 7\cdot10^{-6}$, $\omega_{xz}= 0.2$ arc sec, $\omega_{yz}= 0.3$ arc sec. Such a rotation may be realised as a consequence of both complex elastic deformation and partial plastic one. This problem was solved by the simulation of elastic state of a crystal plate with a quasi plastically deformed area of a shape corresponding to the investigated vicinal sector. The consideration was based on a general solution of the basic equations of the three-dimensional theory of elasticity using known Green functions for infinite body [1]. In usual way this general solution was performed as a sum of two partial solutions: for inhomogeneous infinite body and for homogeneous plate with a properly loading surface. The later part of the problem was solved numerically using Fourier transform. The calculations carried out demonstrate that observed deformation state of the vicinal sector may be a result of pure elastic stresses generated by the quasi plastic deformation of a level $\varepsilon_0= 5\cdot10^{-7}$. It shows that the vicinal sectorial boundaries are coherent and perfect.

The described technique was applied to a number of crystals grown with different rates. Some recommendations about the optimal crystal growth conditions can be formulated.

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X-ray fluorescent microtopography

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X-ray fluorescent microscopy has a number of specific features which give new possibilities in solving traditional problems of microdiagnostics. In this work we consider the problem of the local investigation of a surface relief by an X-ray fluorescent microscope. Two problems are solved -the problem of quantitative description of the signal formation and the inverse problem of the surface relief reconstruction.
It is shown that, under some conditions, the signal equation has the following form:

$$S(x_0) = n_f \int_{x_0}^{\infty} exp\left(-\nu(f(x_0) - f(x)) - \frac{\nu \cos\alpha + \mu}{\sin\alpha}(x - x_0)\right)(ctg\alpha - f'(x))dx$$

where f(x) is the function describing a surface relief, ν is the absorption coefficient of the initial X-ray radiation in material, μ is the absorption coefficient of the fluorescent radiation, nf is the yield of the fluorescence, α is the angle between the direction of the X-ray beam and the direction "sample-detector" (Fig.1).
The solution of inverse problem (i. e. dependence of $f(x)$ from the flourescent signal $S(x)$) is found in form:

$$f(x) = \frac{1}{\nu}\left(\frac{\nu \cos\alpha + \mu}{\sin\alpha}x - \int_0^x \frac{\mu dx}{\sin\alpha(s(x) - n_f)} + ln\left|\frac{S(x) - n_f}{S(0) - n_f}\right|\right)$$

The influence of reqistration signal error on the reconstruction accuracy of the function f(x) is investigated. The results of the reconstraction is presented on the Fig.2. It is shown that the sensitivity of the mthod is proportional $\mu\Delta f/\cos\alpha$ in the vertical direction and $\Delta x(\nu \cos\alpha + \mu)/\sin\alpha$ in transversal direction.

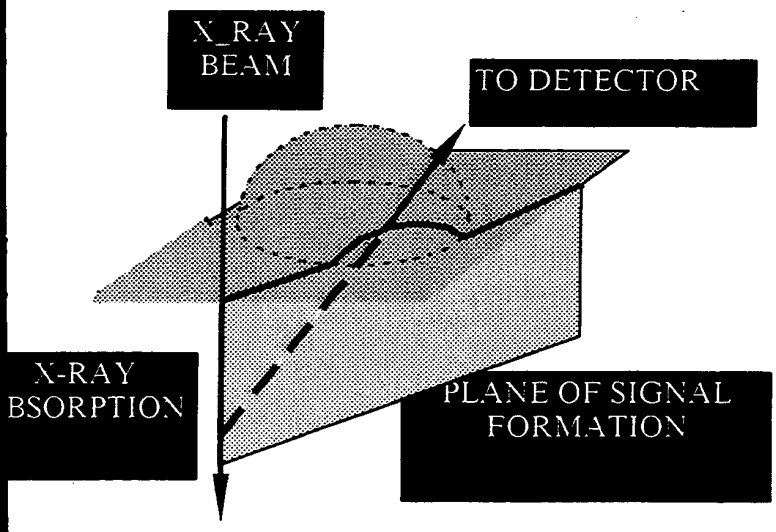

Fig.1.

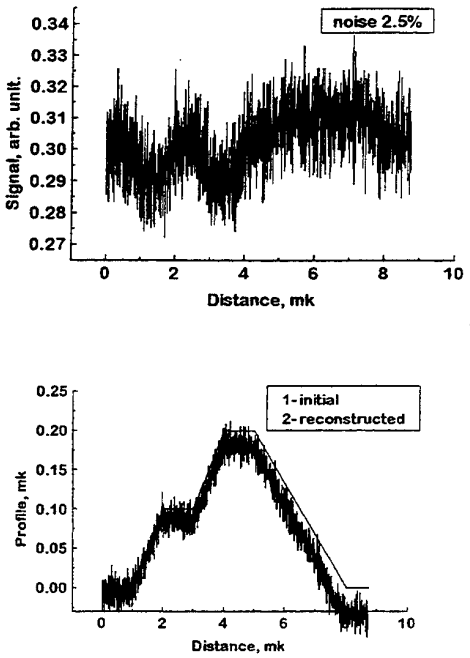

Fig.2 .

EFFICIENCY CALCULATIONS OF COLLIMATOR SYSTEMS WITH INCORPORATED GRADED CRYSTALS AND MULTILAYERS

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Diffraction on bent single crystals or multilayers with laterally graded lattice parameters allows, in principle, to convert divergent x-rays as emitted from a small conventional source into a parallel beam. Therefore, it should be possible to use an x-ray source more effectively. Such systems are also of interest for high-resolution topography and diffractometry. Concerning bulk single crystals, a lattice-parameter gradient could be produced by defined thermal or chemical composition gradients [1]. Whereas the first possibility is low effective, the second could not yet be realized up to now. The use of a multilayer with a laterally graded period combined with a crystal collimator has been described and discussed by Schuster and Göbel [2], who roughly estimated the gain in efficiency. It is the aim of this contribution to give a quantitative theoretical description of various collimator systems with incorporated graded crystals and multilayers including numerically calculated gain factors and other relevant properties.

Generally, the production of broad beams is most effective. Crystals with composition gradients would give the highest gain factors because it is possible to make use additionally of reflection asymmetry. The requirements to be met concerning composition gradients and structural perfection are discussed. Graded multilayers as one-dimensional periodic lattices do not allow asymmetric reflections. It is difficult to compare arrangements with and without multilayers because different geometrical parameters apply for optimum conditions. For a W/Si multilayer combined with a Bartels collimator, with parameters as described by Schuster and Göbel, it yields a theoretical gain factor of 17. For a (hypothetic) graded (Si, Ge) crystal, factors of 21 to 25 would result under the same conditions. Perfect graded crystals, when available, could be used also alone as crystal collimators, e.g., for double-crystal topography gaining intensity by one order of magnitude [(Si, Ge), CuKα, 422 reflection].

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ASYMMETRIC MONOCHROMATOR IN NONCOPLANAR GEOMETRY OF DIFFRACTION

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The problem of obtaining narrow collimated X-ray beams has attracted a widespread attention since the early sixties [1-3]. Narrow X-ray beams can be formed with the aid of a strongly asymmetric reflections with $\beta >> 1$, where β is the asymmetry parameter. Up to now, narrow collimated beams have been obtained only in coplanar geometries of diffraction, where the wave vectors k_0 of an incident beam and k_h of the diffracted beam, the surface normal n_0 and the reciprocal-lattice vector K_h lie in the same plane. A strongly asymmetric geometry is implemented because the crystals are cut out at certain angles and the surfaces are subjected to special thorough treatment. As a rule, such a monochromator can be used only for a definite reflection and a definite radiation wavelength λ.

We have shown that narrow collimated beams can also be obtained using strongly asymmetric noncoplanar geometries of diffraction that allow a continuous smooth variation of the asymmetry parameter over a wide range of its values and the use of different reflections for the same crystal-monochromator. The geometry of such a monochromator and its advantages are discussed.

The CuK_α -radiation passes through a system of slits and is diffracted by the (220) plane forming an angle of $\Psi = 73.22^\circ$ with the monochromator surface S_i cut out parallel to the (211) plane. The variation of the angles of incidence Φ_0 and reflection Φ_h, and therefore, also of β, is attained by rotating the crystal about the reciprocal-lattice vector K_h. The crystal-analyzer was a Si(220) crystal of high perfection with $\beta = 1$ and $\beta = 30$. The geometry suggested is rather simple and universal: it provides the implementation of a limiting asymmetric case of diffraction for any orders of reflections by using standard crystals. The method should be especially efficient for the synchrotron radiation, because in this case there is no necessity to prepare asymmetric crystalline monochromators for a wide range of wavelengths.

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ROTATED-INCLINED MONOCHROMATOR FOR SYNCHROTRON RADIATION: ABERRATION AND ITS COMPENSATION

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Rotated-inclined double-crystal x-ray monochromator was designed for high-power undulator beamlines for SPring-8 to reduce an impinging radiation power density [1,2]. Recently [3] we have shown that an inclined double-crystal monochromator suffers from a certain type of aberration which may be relatively easily compensated. In this paper we show that a similar aberration exists also in the case of rotated-inclined monochromator and that like in the inclined case the aberration may be compensated. This theoretical result is illustrated by ray-tracing.

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X-RAY INSTRUMENTATIION FOR HIGH RESOLUTION EXPERIMENT UNDER MULTIPLE DIFFRACTION CONDITIONS

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We suggest a set of elements united in the modular X-ray spectrometer to easily implement multiple-diffraction layouts with two-dimensional collimation of the incident beam in combination with X-ray standing wave method. The instrument can be used to study single crystals, heteroepitaxial structures, multi-layer systems and superlattices, and crystals subjected to different treatments (mechanical treatment, ion implantation, etc.).

The main elements of the spectrometer are: the unit for generation and monochromatization of the incident X-ray beam, the one-circle goniometer with the vertical axis, the four-circle research goniometer, and different slit systems. All units except the four-circle goniometer can be mounted on two parallel optical guides. The four-circle goniometer is a stand-alone unit consisting of a basic two-circle goniometer with vertical axis of rotation, the chi-circle with horizontal axis of rotation and a small compact torsion-based goniometer mounted on the chi-circle. All rotations are driven by stepping motors via worm gears. On axes where fine (to 0.1 arc sec or less) angular positioning is desirable, torsion-element-based rotation is used; it is driven via piezo drivers. A program package was developed to control the experiments. The software is based on the modular principle providing for fast implementation of application programs suited for specific X-ray diffraction methods.

The instrumentation was tested in the experiments on multiple X-ray diffraction with two-dimensional collimation of the incident beam. The measurements were performed in the (000)(111)(220) configuration on a perfect Ge(111) crystal. For collimation we used two perfect Ge(111) (one flat, the other channel-cut) crystals placed on the goniometers with mutually perpendicular axes of rotation. The collimation in the horizontal direction was determined by the half-width of the flat monochromator, whereas for the (220) reflection it was 12 arc sec. The measurements of the phase-sensitive tails of the rocking curves were performed at different angular displacements from the exact Bragg positioning. The rough theta-positioning was performed with the basic goniometer, the azimuthal rotation of the sample was done with the chi-circle, and the rocking curve measurements were done by the piezoelement-driven rotation of the sample on the compact goniometer. The results of the experiment well compare with the theoretical calculations, which confirms the capabilities of the suggested instrumentation.

TEMPERATURE-GRADIENT CELLS FOR IN-SITU X-RAY TOPOGRAPHY

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X-ray topography is a suitable method for studying structural phase transitions in single crystals. In a crystal plate subjected to a temperature gradient two different phases and their common phase boundary can be recorded simultaneously on one topograph [1-3]. We have developed two different in-situ cells which were used either for conventional Lang-topography or for white-beam synchrotron topography (at the SRS Daresbury, UK).

One cell is a combined heating and cooling device for the temperature range 140K to 500K. It contains two Al rings. The outer ring is hollow and can be cooled by liquid nitrogen, the smaller inner ring is heated by resistance wires connected to a PID temperature controller. The sample is held between Kapton foils which have low X-ray absorption and sufficient temperature stability. A small additional heater near one end of the crystal plate allows to apply variable temperature gradients (Figure 1).

The second high-temperature cell (300K to 1000K) consists of a vacuum chamber with beryllium windows. The sample is mounted between two graphite heater plates which transmit the X-ray beam and produce an almost homogeneous temperature field. This chamber also has an additional heater for variable temperature gradients (Figure 2).

X-ray topographs of transition phase boundaries in NH_4LiSO_4 and $KLiSO_4$, recorded using these cells, will be shown.

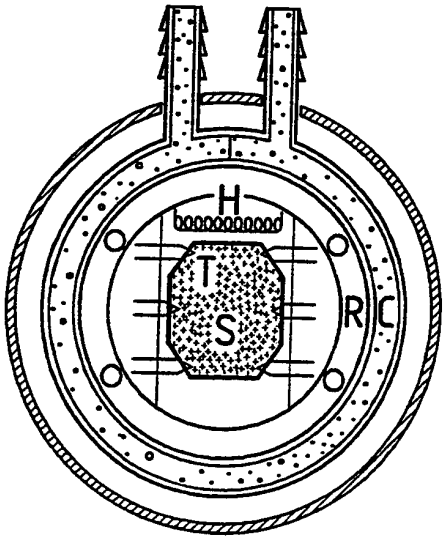

Figure 1: C: cooling ring; R: resistive heating elements; H: additional heater; S: sample plate; T: 6 thermocouples at the periphery of the sample.

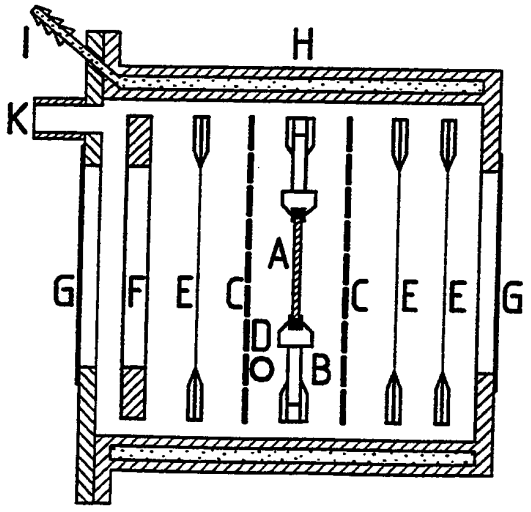

Figure 2: A: sample plate; B: crystal holder; C: graphite heater plates; D: additional heater; E: aluminium foil heat shields; F: ground plate; G: beryllium windows; H: vacuum chamber; I: cooling water inlet; K: to vacuum pump.

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Application of Parabolic, Graded Multilayer Optics in High-Resolution X-Ray Diffraction and X-Ray Reflectometry

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Periodic multilayers are ideally suited as high reflectivity and wide bandpass Bragg reflectors. Their fabrication process allows a lateral variation of the multilayer period and a match to the incidence angle so that for all points of the reflector the Bragg condition can be fulfilled for a selected wavelength. Parabolically curved multilayer mirrors convert divergent radiation of an opening angle of up to 1° emerging from a Cu Kα X-ray source into a parallel beam of about 1mm width and less than 0.03° divergence [1].

In high-resolution diffraction, parabolic multilayer mirrors can be used as condensors to couple the divergent radiation from an X-ray source into channel-cut crystal monochromators [2]. Different configurations consisting of a single parabolic mirror and of a pair of crossed-coupled parabolic mirrors in the Kirkpatrick Baez scheme were investigated. Thereby, about one order of magnitude in intensity and 0.03° collimation perpendicular to the scattering plane of the monochromator were gained. The intensity gain is beneficial for the measurement of extremely thin epitaxial films. The improved collimation has been used to resolve minute lattice mismatches and thickness fringes of thick layers.

The requirements for an X-ray reflectometer are twofold. The ability to measure the range of intensity necessary to characterize the thickness oscillations of atomic monolayers and the coherence to resolve thickness oscillations of about 500nm thick layers. To establish sufficient intensity for measurements on thin layers at higher incidence angles, large samples must be fully illuminated. Parabolic, graded multilayer mirrors enable this full illumination of large samples with a low-divergence "parallel beam".

The layout of parabolic, graded multilayer optics for high-resolution diffraction and reflectometry will be presented. The capability of this novel instrumentation is underlined by applications in high-resolution diffraction, reciprocal space mapping, and reflectometry.

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A High Precision Spectrometer for the Absolute Determination of X-Ray Absorption Edges as Calibration Standards

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The general characteristics and spectrometric features of a high resolution four-crystal reflection x-ray monochromator with wavelength analysis installed at the HASYLAB beamline L at DESY are presented.

The monochromator is part of a spectrometer developed to calibrate x-ray absorption edge spectra in the energy range of 6 - 36 keV with a relative uncertainty $\Delta E/E$ from 10^{-5} to 10^{-6}. This requires an extremely effective suppression of harmonics and also a negligible instrumental influence in order to obtain almost intrinsic spectra. As the results show, the monochromator fulfills the requirements, including very high stability.

One essential advantage of the (+ - - +)-setting is that the monochromator itself limits the divergence of the primary radiation, which is very useful for energies above 5 keV where the natural divergence of the synchrotron radiation exceeds the width of the crystal reflection curve. This setting needs no further optics such as slits to improve the resolution, which is therefore not influenced by vertical movements of the primary radiation source.

Harmonic suppression can be achieved by detuning the channel-cut nondispersive monolithic part slightly out of its parallel-sided position and is described by the detuning angles $\delta_{1,2}$. For reflections at netplanes with *Miller*'s indices all odd, the structure factor of the diamond structure leads to the forbidden harmonic with $n = 2$ in the Bragg-equation $n\lambda_B = 2d \sin\vartheta_B$. It is therefore advantageous to choose reflecting netplanes with odd indices, (e.g. (111) or (311)).

In preliminary tests the operation of the set-up according to the principle was demonstrated. The thermal load of the first crystal caused a temperature increase of approx. 5 K. After reaching temperature equilibrium reproducible results are recieved. In a measured section of 18-30 degrees of arc, the deviation of the monochromator position from that measured by the goniometer is less than the resolution of the monochromator angle decoder. The FWHM of the rocking curves is in the range of the expected values. An example of the calibration procedure for the copper K-edge is given as well as a comparison of the remeasured absorption edge energies with the previously tabulated data.

X-RAY INVESTIGATION OF ULTRATHIN YBCO FILMS FABRICATED BY MODULATED MAGNETRON SPUTTERING

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Abstract

The characterization of the microstructural and morphological properties are of great importance in order to improve the epitaxial growth and the interface morphology of HTc superconductors films. The defect structure, the degree of crystalline perfection and the surface roughness are affected by the initial layer configuration at the early stage of growth.

In this work we report on structural and morphological investigation of ultrathin $YBa_2Cu_3O_{7-x}$ films (YBCO) grown on $SrTiO_3$ substrates (STO). The films are fabricated by inverted cylindrical magnetron sputtering with a modified deposition process which is based on the modulation of sputtering power. This modified deposition technique allows one to fabricate YBCO films with a high in-plane connectivity (grain coesion). The structural analyses of the very thin films (thickness: 10-50 nm) are performed by using low-angle x-ray diffraction (x-ray specular reflectivity and transverse scan), double-crystal x-ray diffraction and reciprocal space mapping close to the (200)/(006), (309)/(303) and (039)/(303) reciprocal lattice points of the substrate/film.

It is well known, that due to the orthorhombic structure of the YBCO bulk material with respect to the cubic structure of the STO substrate, strain field and lattice deformations are strongly involved. Moreover, the crystallographic transition during the deposition process of YBCO epitaxial layer generates dislocations and stacking faults and causes a twinning of the film.

The reciprocal space mapping demonstrate, that in our films grown with the modified deposition technique no twinning of the structure occurs if the film thickness is below the critical thickness (<15 nm). Furthermore, double-crystal diffraction measurements and reciprocal space mapping reveal that the films are tetragonally distorted. Specular reflectivity measurements show distinct Kiesseg fringes revealing a well defined film/substrate interface and a smooth surface. The surface roughness is investigated by x-ray transverse scan measurements and atomic force microscopy.

INTERFERENCE AND DIFFRACTION INVESTIGATIONS OF POROUS SILICON DEFECTS BY X-RAY TOPOGRAPHY

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The contrast particularities arising in the topographs of the epitaxial heterosystem of Si/ porous Si/ (001) Si substrate are discussed. The double crystal X-ray topography in the back-scattering geometry has been used. The X-ray rocking curves of the different reflections exhibit the same peak position for the substrate and the epitaxial layer, while the peak of the porous Si is remarkably shifted into the region of greater Bragg angels. The most typical defects of these systems observed in the porous Si peak topographs are the dark roundish patches with black or black-white perimeter mounts depending on the value of a deviation from the exact Bragg position of the crystal.

The interference contrast is obtained for topographs registered using the asymmetrical (311) reflection, when the angle between the incident beam and the (001) crystal surface is only 2.8^{o}. Three types of the black-white interference contrast patterns can be observed. They are the nearly rectilinear periodic fringes along the sample edges (1), the concentric rings (2) and the large indistinct quasiperiodic changes of the contrast across the sample surface (3). The analysis of the diffraction and interference contrast behavior peculiarities depending on the registration conditions gives us the opportunity to explain the nature of the mentioned defects.

It is shown, for the first time, that the studied epitaxial system is a Bragg-case interferometer, in which the porous Si layer acts as an air gap in ordinary X-ray interferometer. The possibility of the interference topography for the investigation of layered systems used in the semiconductor technology are discussed.

White-beam synchrotron topographic investigation of tungstate flux grown $KTiOAsO_4$ crystals

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Abstract

White-beam synchrotron radiation topographic investigation of non-linear optical crystal $KTiOAsO_4$ (KTA) has been carried out. The crystals were grown from tungstate fluxes. It is shown that growth striations are primary planar defects. By selecting the wavelength to activate anomalous scattering effect near absorption edge of atom As, antiparallel ferroelectric domains are revealed. In reflection pairs of (hkl) and (hkl), the domain contrast demonstrates reversing and the ratios of $|F(hkl)|^2$ to $|F(hkl)|^2$ are calculated. The domain configuration with zig-zag walls are also discussed in terms of the structural characteristics of $KTiOAsO_4$.

High resolution X-ray diffraction study of the Bragg peak width in highly mismatched III-V heterostructures

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Abstract

Many investigations have been performed to study the lattice strain and the extended crystal defects in highly mismatched heterostructures. Recently, the possibility to use X-ray diffraction to measure the threading dislocation density has been proposed by Ayers and co-workers [1,2]. The method is based on the analysis of the Bragg peak width. The dislocations broaden the Bragg peak through two different mechanisms: they induce a rotation of the crystal lattice (tilt effect and mosaicity) and modify the lattice parameter of the crystal close to the dislocation line (strain effect). According to Ayers model, the two components can be separated by comparing the layer Bragg peak width at different reflections.

In the present work, the results of high resolution X-ray diffraction experiments performed on (001) oriented InAs/GaAs, GaAs/InAs and highly mismatched InGaAs/GaAs heterostructures are reported and discussed. The thickness of the samples is of the order of 2 μm or more. Different geometrical conditions and reflections, including the symmetric 004 and the asymmetric 115, 335, 444 and 117, have been investigated.

For a given asymmetric reflection the grazing incidence geometry always produced much narrower Bragg peaks. To explain such an effect it is necessary to assume that lattice tilt and strain have some degree of correlation. Reciprocal lattice maps measurements performed on the heterostructures are similar to those reported by Heinke at al. [3]. The interpretation of the experimental results in term of layer surface roughness or elastic strain relaxation is discussed.

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X-RAY CHARACTERIZATION OF AN ESAKI-TSU SUPERLATTICE FOR TRANSPORT INVESTIGATIONS

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By X-ray investigations we studied structural parameters of a MBE grown Esaki-Tsu superlattice structure that exhibits in transport measurements a negative differential conductance. We determined the structural parameters by X-ray diffraction and X-ray specular reflectivity measurements. We are especially interested to study the influence of the additional thickness graded layers embedding the GaAs/AlAs superlattice that was introduced for a carrier confinement.

According to Esaki and Tsu [1], an ideal composition superlattice (SL) is formed by the periodic arrangement of two layers of different materials separated by abrupt interfaces which results in a splitting of the energy bands into minibands and minigaps. To investigate the miniband transport in a superlattice structure a carrier confinement between the SL and the bulk GaAs is necessary. Our SL (108 periods; each consisting of 11 monolayers of a GaAs well and of 7 monolayers of AlAs barrier; continuously doped with silicon) was embedded between two symmetrically arranged thickness graded layer substructures [2,3] delivering an adaptation of the band structure. The grading begins at the GaAs substrate or cap layer, respectively, the well thickness remains constant and the barrier thickness increases towards the SL. The structural investigations were performed by wide angle, by high resolution X-ray diffraction measurements of the fundamental (002,004) Bragg reflection, and by specular X-ray reflectivity measurements close to the (000) reflex at small angles. The presence of the thickness graded layers leads to a asymmetric broadening of the satellite peaks and to an additional modulation between the low angle first and third satellite peaks. The current-voltage characteristic of the lateraly structured SL showed an Esaki-Tsu like current-voltage characteristic and was almost ohmic at small voltages. The ohmic behavior for small voltages indicates a well adopted band structure. The analyzis of this thickness graded layers give us the possiblity to optimize the structural and transport parameters of the SL and to control the growth process.

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STRUCTURAL CHARACTERIZATION OF InAs / GaSb SUPERLATTICES

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InAs/GaSb superlattices (SLs) are of current interest for infrared optoelectronic detector applications. We studied InAs/GaSb SLs with InAs layer thicknesses ranging from 4 to 14 monolayers with the thickness of the GaSb barriers kept constant (10 monolayers). Two series of samples with different interfaces were grown on undoped 2-inch (100) GaAs wafers by solid-source molecular beam epitaxy using a valved cracker As-cell and a standard cracker Sb-cell supplying molecular beams of As_2 and Sb_2, respectively. Depending on the shutter sequence within the superlattice either InSb-like or GaAs-like interfaces were established. Prior to the deposition of the superlattice a strain relaxed GaSb buffer layer was grown. Structural characterization has been performed using high-resolution X-ray diffractometry, Fourier transform far infrared spectroscopy and Raman scattering.

The high structural quality of the InAs/GaSb superlattices is confirmed by strong X-ray superlattice peaks and Raman spectra showing backfolded acoustic phonons up to the 7^{th} order. One- and two-dimensional X-ray reflection profiles (angular space maps) show significant differences in the structural perfection of the superlattices depending on the type of interface bonds formed between the InAs and GaSb layers.

X-ray diffractometry with symmetric and asymmetric reflections enabled the measurement of lattice parameters of buffer layers and superlattice films parallel and perpendicular to the growth direction. Using profile simulations a detailed account of the strain situation within the SLs was obtained. Samples with 6 and more InAs monolayers and InSb-like interfaces are elastically strained to the lattice constant of the underlaying relaxed GaSb buffer. Samples with 4 InAs monolayers and those with GaAs-like interfaces are partially relaxed. Strain profiles throughout the SL stacks of these partially relaxed samples have been obtained via modelling of the reflection profiles.

Samples with InSb-like interfaces show a higher degree of perfection than those grown with GaAs-like interfaces where we observe considerable peak broadening especially with Ω-scans. From the evaluation of Ω-scan and $\Theta/2\Theta$-scan profiles of 00L reflections the various contributions to the observed peak broadenings are separated. Mosaicity within the superlattice is the main contributor to the peak broadening observed with Ω-scans.

INVESTIGATION AND CALCULATION OF DISLOCATION CONCENTRATION IN MASSIVE QUARTZ CRYSTALS

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The investigation of dislocation density in massive quartz crystals was carried out with the help of γ-ray diffractometer. High monochromatic γ-ray radiation from ^{198}Au source activated by nuclear reactor neutrons (E=412 keV; λ=0,03 Å; $\Delta\lambda/\lambda \leq 10^{-6}$) was used. The beam cross section was $0,1 \times 10$ mm^2 with angular divergence of 9 angular seconds.

The angular distribution of intensity and the integral reflectivity coefficients (Ri) from crystallographic planes $(1\bar{1}00)$ and $(11\bar{2}0)$ in Laue-geometry were measured. For investigation of the different regions of crystal the sample have moved normally to the γ-ray beam.

Although strictly mathematically the problem of dynamic X-ray diffraction at dislocational crystals is not yet solved, a number of models are developed enabling to calculate dislocation concentration in crystals and well agreed with the experimental results in the case of X-ray diffraction. Our preliminary investigations have shown that in the case of γ-ray diffraction such approach is limited by the large value of extinction length Λ_o for shortwave γ-ray radiation.

The most promising seems the method derived from statistical dynamical diffraction theory by N. Kato, where no preliminary assumptions about the structure of crystal's defects are made. In this theory the experimental diffracted beam intensity is considered as dynamic intensity, being averaged over statistical field of crystal's distortions.

The comparison data were obtained for several quartz crystals on determination of dislocation density by various means: with using γ-ray diffractometer, X-ray topography and by calculation of etching channels in AT-cut plates for the same crystals.

Good agreement of the results obtaining by these methods shows perspectivity of γ-ray diffraciometry method for non-destructive dislocation concentration control in massive quartz crystals.

HRXRD and ACT Study of Si-doped GaAs.

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Silicon doped (n-type) GaAs substrate wafers are commonly used in micro-electronic device applications requiring conducting substrates. The lattice parameter for commercially available GaAs (Si, n-type) crystals can not be assumed a priori. Equal amounts of Si dopant have been separately reported to both expand and contract the lattice parameter of the GaAs. Small uncertainties in substrate lattice parameter can have a significant effect on the x-ray diffraction techniques used to calculate the composition of hetero-epitaxial layers. Variations in the substrate lattice parameter produce the greatest error for systems, such as AlGaAs/GaAs, which have a small overall mismatch. A change in the GaAs substrate lattice parameter as small as 0.00008 Å can introduce a 1%Al error in the calculation of Al content in AlGaAs on GaAs.

This study surveyed several commercially available GaAs (Si, n-type) substrate wafers prepared by: horizontal Bridgeman (HB), vertical Bridgeman (VB), vertical gradient freeze (VGF) and liquid encapsulated Czochralski (LEC). SIMS measurements confirmed that the Si dopant concentrations for all the samples were in the 1.2 - 3.5 x 10^{-18} cm^{-3} range. The lattice parameter of the GaAs(Si) substrate was measured relative to a thick layer of undoped, epitaxial GaAs that was used as the reference. High resolution x-ray diffraction (HRXRD) was used to measure the lattice parameter difference between the GaAs epitaxial layer and the GaAs(Si) substrate. Differences in lattice parameter were measured from rocking curves of the (006) reflection. The angular separation between the epilayer and the substrate can be determined with a precision of 1-2 arc seconds, corresponding to a difference in lattice parameter of ~0.00001 Å.

The VB and HB GaAs(Si) samples had a lattice parameter that was ~0.0001 Å larger than semi insulating GaAs while the VGF and LEC samples had a lattice parameter that was ~0.0002 Å smaller. The lattice parameter variations between Bridgeman and VGF methods were greater than either the boule to boule or supplier to supplier variation for the same growth technique. Asymmetric crystal x-ray topography (ACT) highlighted fundamental differences between the Bridgeman and VGF materials. Topographs of VB and HB samples, with an EPD count of ~2000-5000 cm^{-2}, showed a faint cellular dislocation network related to threading dislocations. Topographs of VGF samples, which had EPD count of 100-500cm^{-2}, showed only a swirl pattern commonly associated with small amounts of segregation at the growth front. Topographs of LEC samples showed extensive crosshatching probably caused by the presence of precipitates. Polaron measurements of the carrier concentration, Hall measurements of carrier mobility and additional SIMS measurements of possible impurities were also used to determine the Si content and degree of activation in these substrates.

THE X-RAY TOPOGRAPHY INVESTIGATION OF DEFECTS IN ERBIUM AND THULIUM DOPED $LiNbO_3$

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Lithium niobate is the material of considerable interest in view of its unusual ferroelectric optical, acoustic and piezoelectric properties. Recently we notice the progres in growth technology of $LiNbO_3$ with rare earth dopants. That offers the possibility of obtaining crystals of modified properties suitable for using in new applications e.g. in laser technology. In this new applications good quality is of great importance.

In the present work we performed the investigations of some new type of lithium niobate crystals by means of Lang X-ray topography, in particular the $LiNbO_3$ Czochralski grown crystals heavily doped with erbium 0.1 - 0.24% at and thulium 0.3- 1.2 %at. The Lang topographs were compared with microscopic obserwation of each patterns.

It was stated that the crystal with erbium concentration close to 0.24% contained low density of dislocation and other defects on the level of $100/cm^2$. In this case it was possible to perform identification of dislocations using Lang method. A number of dislocations have been identified on the base of topographs in reflections from sets of equivalent lattice planes e.g. $(\bar{2}110)$, $(1\bar{2}10)$, $(11\bar{2}0)$, $(1\bar{1}02)$, $(\bar{1}012)$, $(01\bar{1}2)$, The dislocation of two types of Burgers vector $1/3\langle 1101\rangle$ and $1/6\langle 2201\rangle$ were found. The present topographs revealed also certain concentration of small precipitates and in some cases boundaries of ferroelectric domains.Besides the dislocations and domains boundaries were revealed by selective etching.

$LiNbO_3$ crystals doped with thulium contained lower density of dislocations to compare to undoped $LiNbO_3$ too, especially on the $LiNbO_3$ 0.6 %at Tm sample - but there are more of different precipitates and even twins. The lowest density of dislocations we nottice in the $LiNbO_3$ doped with erbium and thulium simultanously. But the growth striations and some of precipitates in the central part of samples have been observed.

HIGH RESOLUTION X-RAY DIFFRACTION AND TOPOGRAPHY OF MERCURY MANGANESE TELLURIDE EPITAXIAL LAYERS

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The crystal perfection of mercury manganese telluride (MMT) films grown by metalorganic vapour phase epitaxy (MOVPE) has been investigated using high resolution x-ray diffraction (HRXRD) and synchrotron topography. The epitaxial films have been grown on (100) GaAs substrates with CdTe and ZnTe buffer layers, using the interdiffused multilayer process (IMP), where alternate layers of HgTe and MnTe are deposited to form the ternary compound.

This growth technique has been found to produce layers of reasonable uniformity in composition across the layer, as seen using double axis diffraction positional maps. Synchrotron double axis topography, using both symmetric and asymmetric reflections which are sensitive to out-of-plane and in-plane strains respectively, have also shown the layers to be of high quality.

Double axis diffraction scans have shown a slight residual strain in the 1μm thick CdTe buffer layer, of the order of 1%. MMT layers show a variation in the rocking curve peak position which could be caused by strain or a variation in manganese composition and hence lattice parameter. In order to resolve these, quasi triple axis diffraction reciprocal space maps were taken at Daresbury Laboratory using slit collimation to give an analyser acceptance angle of 15". This method provides moderate resolution with a reasonable counting time due to the high intensity.

Symmetric and asymmetric scans show thick (6μm) layers of MMT to be fully relaxed, but some residual strain was present in the thinner layers. Thick layer θ-2θ scans show a marked shoulder in the MMT peak which corresponds purely to lattice parameter variations. From the variation of intensity with scattering vector we deduce a change in concentration with depth in the crystal.

X-RAY STUDIES OF CRYSTAL PERFECTION IN DISTORTED DIAMONDS

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Some diamonds from the Argyle Diamond Mine, of good morphology but distorted interiors, have been examined by synchrotron X-radiation and other methods. This single mine produces one-third of the world output of natural diamonds and is especially noted for its pink diamonds. Many of the brown diamonds appear to have experienced plastic deformation, have high dislocation densities and have been reported to offer high abrasion resistance. Diamonds comprising brown and white halves were selected for the present study, the boundary being approximately parallel to a (111) plane in each specimen.

The 800 symmetric reflexion from a silicon monochromator was used to match the diamond 333 reflexion, selecting a wavelength of 0.096 nm in pure σ-polarization from the synchrotron spectrum at the Daresbury Laboratory (Warrington, UK). Rocking-curve widths were measured as 280 arc seconds for the brown regions and 75" for the white parts. Compare these values with 3", the rocking-curve width calculated from dynamical theory for a perfect diamond in this diffraction geometry. These large rocking-curve widths are attributed to considerable lattice strains.

Whole-crystal rocking curves have shown that the brown and white regions have a relatively large angular separation, typically 0.35°. By varying the specimen-to-film distance and by studying the consequent displacement of prominent features in double-crystal topographs, it has been deduced that the divergence of the doubly-diffracted beam was of the order of 0.1°, confirming the extraordinary 'lattice curvatures' of these natural diamonds. Single-crystal (white-radiation) topographs showed 'plumes' of reflexion caused by curled regions of crystal diffracting the various x-ray wavelengths over wide angular ranges.

Fourier-transform infra-red spectroscopy indicated that the brown parts contained pairs of nitrogen atoms (A aggregates) and some {100} platelets of average radius 35 to 40 nm; whereas the white parts contained B aggregates (four N atoms surrounding a vacancy) and platelets of average radius 5 to 12 nm. The nitrogen concentrations for the brown and white parts have been calculated to be approximately 80 parts per million and 400 ppm respectively: surprisingly much more nitrogen in the white parts than in the brown.

Argyle diamonds are often highly distorted and the brown colouration is likely to be the result of plastic deformation in conjunction with defects. The brown-and-white diamonds studied here certainly have highly strained structures: more so in the brown than in the white parts. An interesting speculation is that the aggregation of nitrogen might relieve strains.

X-RAY DIFFRACTION STUDIES OF SYNTHETIC SINGLE-CRYSTAL DIAMONDS

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Large single-crystal synthetic diamonds, grown by the reconstitution technique from seed crystals, are used in number of industrial applications, such as turning tools and wire-drawing dies. They are usually grown in either the [111] or the [001] directions, and have approximately hexagonal or square cross-sections respectively. Diamonds 2 mm x 2 mm x 1 mm are typical, but larger sizes can be grown. Three (111) specimens and four (001) were studied here by x-ray topography, using both conventional and synchrotron sources of x-rays.

The Synchrotron Radiation Source (SRS) at the Daresbury Laboratory (Warrington, UK) was used for both single-crystal (white-radiation) and double-crystal (monochromatic) topography to image growth-sector boundaries and dislocations, as well as any metal inclusions. Single-crystal topographs showed some x-ray interference fringes associated with the strain fields around inclusions and growth-sector boundaries, which can be described by the dynamical theory of x-ray diffraction.

For double-crystal topography of the (111) specimens, the 800 symmetric reflexion from a (100) silicon monochromator was matched with the 333 symmetric reflexion from the diamond. The monochromator was set at a Bragg angle of 45° to select radiation of wavelength $\lambda = 0.096$ nm (and its harmonics) in pure σ-polarization, in a convenient and non-dispersive diffraction geometry. Since the 666 reflexion is 'forbidden' in diamond, the $\lambda/2$ harmonic makes little contribution; and $\lambda/3$ and higher orders were very weak in the synchrotron spectrum used. The resulting topographs and rocking curves were therefore taken in almost monochromatic radiation, in which the calculated rocking-curve width is 3 arc seconds for a perfect diamond. All four azimuthal orientations (χ = 0, 90, 180 and 270°) were used for the topography and rocking-curve analysis, in order to separate effects due to lattice parameter variations from misorientations. Here typical rocking-curve widths ranged from 10" to 20".

The specimens studied showed a wide range of crystal perfection and a variety of defects. Topographs demonstrating the various types of imperfection will be shown.

THE EFFECT OF PARTICLE INCLUSIONS AND IN-SITU CRYSTAL DAMAGING ON THE FACE GROWTH RATE AND CRYSTAL MORPHOLOGY.

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The face growth rate of crystals and thus their morphology is decisively influenced, among others, by the real structure of the crystals. In this work the formation of dislocations by intentionally induced in-situ perturbations of the growing faces and its effect on crystal growth is studied quantitatively combining in-situ optical observations (reflection microscopy and laser Michelson interferometry) with subsequent X-ray topography (Lang technique).

Another aim is to determine the types (Burgers vectors) of dislocations formed on different growth faces and correlate them to the observed surface patterns and normal growth rates of the faces. Experiments were performed with organic crystals (salol, benzophenone) growing from slightly supercooled melt and with potassium alum crystals growing from aqueous solution.

Two kinds of face perturbations were introduced:

- A small tin ball (0.5 - 1.5 mm in diameter) was placed on a almost perfect growing face and incorporated.
- The relatively undisturbed growing face was slightly scratched with a fine needle.

In-situ reflection microscopy of the perturbed face segment did not show an essential change of the surface morphology in the first period after deposition of the tin ball until the moment the ball was just overgrown and incorporated. In this moment fast and drastic changes of the surface pattern occurred, developing growth hillocks and more or less irregular macroscopic growth steps moving with some millimeters per minute across the face. The same fast change of the surface pattern appeared immediately after scratching the growth face. A series of growth hillocks developed along the scratch, indicating the generation of a row of dislocations by the mechanical damage. By laser interferometric measurements the heights and slopes of the growth hillocks and the face normal rates were determined. Due to the dislocations induced by particle inclusions and in-situ scratches the growth rates of the involved face were increased by a factor of up to 3.

For X-ray topography plates (1 - 2 mm thick) containing the intentionally introduced perturbations (particle inclusions or scratches) in the region "behind" them (viewed in direction of growth) were cut out of the crystals. The topographs show extended strain fields surrounding the inclusions, obviously due to the different thermal expansion of crystal matrix and inclusion. From the closure side of the inclusions and from the scratches numerous dislocations originate. They extend with growth, ending roughly normal on the growth face involved in the perturbation. In some cases the local density of dislocations is so high that their topographic images cannot be resolved and the Burgersvector determination is difficult. The X-ray topographic results are correlated to the optical surface observations and are discussed.

REAL STRUCTURE FORMATION FEATURES OF LEC GROWN SEMI-INSULATING GaAs SINGLE CRYSTALS

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At present semi-insulating GaAs single crystals are considered to be the most promising material for building a number of microelectronic devices, and high speed integrated microcircuits, in particular. In this connection the problem of uniformity of crystals grown is assuming considerable importance. It has been established by numerous investigations that the basic electrophysical parame-ters of semi-insulating GaAs (such as deep donor EL-2 distribution and concen-tration, resistivity, Hall's mobility and others) correlate with density and distribu-tion of structural defects, of dislocations in particular. Thus, electrophysical pa-rameter uniformity increase is directly associated with the improvement of struc-tural characteristics of the material.

In the present work investigation has been carried out concerning formation peculiarities of the real structure of LEC-grown semi-insulation GaAs single crystals with large (60-80 mm) diameter. Various low-energy effects on crystallization process (ultrasonic vibration, low-frequency vibration, non-periodic oscillations of a crystal and a crucible) were used to improve the uniformity of crystals. Such outside effects are highly efficient in single crystal growth [1], but, however, the optimum conditions of using them and the nature of their impact on the real structure of crystals grown have not been studied in full measure.

GaAs single crystals doping by Si and Te were used for outside effects optimization.

Using the methods of doublecrystal X-ray diffractometry, topography and metallography we obtained data on structural and impurity nonuniformities in such crystals and on their evolution while changing the parameters of influence. To obtain detailed information on dislocation formation and their distribution the dislocation structure has been studied in {001}, {110} and {111} wafers cut from various parts of a boule. It has been established that it is typical far crystals grown to have the combination of two dislocation configuration types: the typical cell form distribution and the slip bands whose formation indirectly testifies to incomplete low temperature relaxation suppression. Some characteristic features of dislocation formation and their distribution near the seed region as well as in the major part of crystals have been revealed by means of structural investigations.

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Studies on Structure and perfection of 4-Aminobenzophenone (ABP) Crystalline thin films using X-ray diffraction techniques.

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Crystalline 4-Aminobenzophenone(ABP) is one of the best non-linear optical materials because of its short cut-off wavelength(at 410nm) and high optical damage threshold (350GM/cm^2, 1064nm, 30ps). It can also be phase-matched to output SHG blue light with a high d_{eff} of 50.3 p/mv at the fundamental wavelength of 870 nm, which is one of the biggest d_{eff} values found in currently available organic crystals. Due to the advantages of organic optical waveguides over bulk crystals in integrated optics applications, many organic crystals, for example mNA, MNA, DAN and MBA-NP have been fabricated into single crystalline thin films. We have obtained some ABP thin crystalline films from vapour, melt and solutions. Structure and perfection assessments are being carried out using X-ray diffraction techniques in our laboratory and Daresbury laboratory. Some information about the orientation of these grown films has been obtained. On the basis of this information, some experimental parameters have been improved and some ABP thin films with good orientation and optical qualities have been obtained.

CHARACTERIZATION OF SPINELS, RUBY, SAPPHIRE AND COLOURLESS CORUNDUM BY X-RAY TOPOGRAPHY

by

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The X-ray topography- Lang method- has been applied to study materials interesting from the industrial and, in particular, gemmological point of view. The studied samples were boules of spinels, ruby, blue sapphire and colourless corundum industrially grown by the Verneuil method. This technique is of particular interest to the industry because it does not need crucible and because it allows to grow large samples at low cost. Nevertheless, the crystals show a very distorted crystalline lattice as demonstrated by the anomalous birefringence on the spinels and by the 2V variation shown by different zones of a given boule of corundum.

In our case, several sections of a boule were cut parallel or perpendicular to the growth axis. The different sections, corresponding to different growth stages, have been studied by optical microscope and by X-ray transmission topography- Lang method-. We could therefore show that the crystal quality is, in all the studied cases, different in different parts of a same boule. In particular, from the X-ray topography it could be pointed out that:

a) the boules of spinel are polycrystalline but with large areas showing the same topographic contrast, therefore monocrystalline, in the peduncular or near peduncular zones. The apical parts are always made by small crystals tilted one to another by small angles.

b) Among all the varieties of Verneuil corundum (colourless, ruby or blue sapphire) the last ones show a better crystalline quality and they often result monocrystals in the lowest or central parts of the boule.

c) On all the samples of corundum a better crystalline quality in the central parts of the sample with respect to the periferical ones has been observed.

All the features have been related to the growth conditions of the different parts of a boule and to a possible rearrangement of the lattice when the sample is moved down from the flame during the growth.

X - RAY TOPOGRAPHIC STUDY OF DEFECTS IN < 100 > AND < 111 > GROWTH SECTORS IN A NATURAL DIAMOND.

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Diamond is subject to intensive research in Materials Science and in Earth Science, consequently it has been studied by a wide range of techniques. Among these, X-ray diffraction topography play an important role giving a large-scale view of low defect density crystals. Structural defects are in fact important for both mineralogical and technological applications because they contribute to the reconstruction of the growth and post-growth histories and at the same time they can be responsible for modifications of the material properties.

The aim of this work was a further investigation by X-ray diffraction topography of structural defects of a natural diamond from Central Yakutia (Russia), as a first necessary step towards the characterization of natural diamonds as materials for electronic devices.

X-ray topographs revealed the sequence of four growth stages during which periods of mixed-habit growth occurred. During the first growth stage only cubic sectors grew and a few mixed type dislocations were nucleated. In the second stage both cubic and octahedral sectors were observed: edge dislocations and "area defects", bounded by curved surfaces, characterize the cubic sectors. In the third stage, octahedral faceting occurred in the (001) cubic sector. In the last growth stage only octahedral sectors developped.

By comparison with experimental work on synthetic diamond, it will be shown how the studied natural crystal recorded variations of the chemio-physical conditions of the growth medium. In fact "area defects" are related to ehnanced absorption of impurities in the cubic sector - the so-called chemical sector zoning - whereas their sequence is related to the chemical evolution of the growth medium - intrasectoral zoning; their curved boundaries and faceting are related to P, T variations of the growth solution.

Determination of the Deformation State of HgSe/ZnTe layers

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For layers thinner than a critical thickness, the cubic unit cell of the epilayer is expected to accomodate the full mismatch by elastic deformation resulting in a tetragonal distortion of the unit cell. When the layer thickness exceeds the critical value, relaxation of the strained epilayer occurs. Normally it is assumed that in the case of (001) orientation the unit cell of the layer remains tetragonal. For II-VI materials it was reported that this simple model is not valid [1]. Any deviation from tetragonal symmetry may influence the determination of the unstrained lattice parameter and the degree of relaxation. A buffer layer with a lattice parameter different from that of substrate and second layer causes a further complication. Therefore, the exact knowledge of the deformation tensor of each layer is necessary.

For the system HgSe on a thick ZnTe buffer layer deposited by MBE on GaAs substrate, the six components of the reciprocal metric tensor of each layer were determined by a least-squares algorithm from a set of reciprocal lattice vectors ({ 1 3 7 }, { 5 5 3 }). The interplanar spacings were measured by the Bond method using CuKα_1 radiation and a Bartels monochromator. From the metric tensor, the deformation tensor and the cubic lattice parameter of the relaxed layer material are calculated assuming the strain components perpendicular to the layer surface to be zero. A principal-axis transformation of the calculated deformation tensor shows that the deformation in the interface plane nearly parallel to the [1 1 0] and [1 $\bar{1}$ 0] directions differs by more than 15 %. This difference is a function of the epilayer thickness and reaches a maximum for layers slightly thicker than the critical thickness.

It is assumed that similar problems occur in other II-VI materials.

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STRUCTURAL PROPERTIES OF III-V HETEROSTRUCTURES STUDIED BY HIGH RESOLUTION X-RAY DIFFRACTION

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The multilayers are of great interest due to their unique optical and electrical properties and their numerous applications in modern high speed communication technology.

The layers, mostly $Ga_{1-x}Al_xAs$, were deposited by molecular beam epitaxy and metal-organic vapour phase epitaxy on *GaAs* (100) substrates. Experimental data are obtained using the nearly parallel setting of X-ray double crystal spectrometer. For this purpose new Ge monolithic beam conditioner was designed and realised. High power X-ray generator Rigaku 300 was employed. Experimental diffraction curves exhibit different oscillation periods due to the total thickness of the layer structure and the thickness of the cap layer. The angular positions of the most intensive subsidiary peaks yield the information about the composition of the individual layers. The quality of substrates was checked by X-ray topography.

Strain depth profiles of the layer systems investigated were achieved with the help of diffraction profile simulations using the dynamical X-ray diffraction theory for distorted crystals. Real interface imperfection and/or composition fluctuation can cause a disagreement between experimental and theoretical curves. For the selected examples of heterostructures and superlattices the lattice mismatch, number and thickness of individual component layers and the superlattice period are given. The results are related with the information given by photoluminescence and Raman scattering, too. The structural parameters were continuously compared with the growth conditions and helped to improve the technological process conditions.

The new monochromator used gives parallel beam defined well enough. The fit between experimental and simulated rocking curves were satisfactory. The theoretical curves calculated on pure dynamical theory basis were compared with commercial program RADS and good agreement was found.

The attempts to use three crystal diffractometer method were also performed.

RECIPROCAL SPACE MAPPING ON $Si_{1-x}C_x$ EPILAYERS AND $Si_n/C/Si_n$ SUPERLATTICES

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Recently it has been shown that with growth techniques like molecular beam epitaxy carbon can be incorporated substitutionally in the Si lattice with concentrations significantly above the bulk solubility limit of $3.5x10^{17}$ cm^{-3}. Such $Si_{1-x}C_x$ layers are of interest because of their potential for applications in Si based heterostructures. We have investigated MBE grown $Si_{1-x}C_x$-epilayers ($x<0.02$) on (001) Si substrates (growth temperature 480°C) as well as $Si_n/C/Si_n$ superlattices ($20<n<60$) with periods in between 10 to 100. Using high resolution x-ray diffraction (triple axis diffractometry) reciprocal space maps around the (004) and (224) reciprocal lattice points (RELPs) were recorded. From the reciprocal space maps it points out that the $Si_{1-x}C_x$ epilayers with layer thicknesses below 150 nm are pseudomorphic in the as-grown state. The superlattices with one monolayer (ML) of carbon[1] grow pseudomorphic as well as long as the number of periods is less than 20 for n=20 Si layers[2]. With increasing n, for pseudomorphic growth the number of periods can be increased as well. Due to the large lattice mismatch between carbon and silicon (larger than 50%) the strain relaxation in the $Si_{1-x}C_x$ alloys differs from that in $Si_{1-y}Ge_y$ alloys[3]. We have annealed the as grown $Si_{1-x}C_x$ at temperatures of 840°C for time intervals ranging from 15 minutes to several hours. In the reciprocal space maps the RELPs corresponding to the $Si_{1-x}C_x$ layers show a shift towards that of the Si-substrate, indicating a decrease of the substitutional incorporation of carbon. However, the layer remains fully pseudomorphic. Furthermore, the RELPs of the annealed $Si_{1-x}C_x$ layers exhibit also remarkable diffuse scattering along the omega direction. Indeed, these findings support earlier suggestions for the nucleation of SiC precipitates and their diffusion controlled growth during the annealing experiments. Similarly, the RELPs of even as-grown $Si_n/C/Si_n$ superlattices exhibit apart from the coherent scattering also diffuse contributions if the number of periods is too large or the number of Si-ML (n) is too small. Also pseudomorphically grown Si/C/Si superlattices exhibit diffuse x-ray scattering after annealing steps. Similarly to the $Si_{1-x}C_x$ epilayers the carbon content in the superlattices decreases during annealing. These x-ray investigations give information on the strain stabilization in Si/C/Si superlattices with high carbon contents as well as on the transition region where precipitation occurs. This behavior is completely different from Si/SiGe/Si superlattices where annealing causes relaxation through misfit dislocations as evidenced by strain relaxation in the reciprocal space maps. The x-ray data on the $Si_{1-x}C_x$ epilayers and Si/C/Si superlattices were analysed not only with respect to the extreme of the coherent scattering, but also an attempt was made to simulate the diffuse scattering on the basis of model calculations considering small defects[4].

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HIGH-RESOLUTION DOUBLE CRYSTAL X-RAY DIFFRACTION DETERMINATION OF THE STRAINED STATE IN SURFACE-LAYER STRUCTURES OF ARBITRARY CRYSTALLOGRAPHIC SYSTEM

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With advances in the epitaxial growth techniques and due to the importance knowing the structural characteristics of the epitaxial layers for application in high-performance electronic and optoelectronic technology the high-resolution double crystal X-ray diffraction (DCXRD) has quickly become a most essential and versatile tool for relatively fast and nondestructive characterizing heteroepitaxial structures. However, in most applications the information capability of the rocking curves is not sufficiently used. In general, the task of strain state description in surface-layer structures is to determine all the components of deformation (or distortion) tensor.

In the present work we propose a new method to determine the strained state in surface-layer structures of arbitrary crystallographic system using double-crystal X-ray diffractometry. In order to analyze and interpret the experimental DCD data we derived new formulae which relate the differences in Bragg diffraction angles obtained on both symmetric and asymmetric rocking curves and the distortions of the unit cell of arbitrary crystallographic system. Our model consider the most general deformation of the general triclinic unit cell. The possibility to calculate all six components of the strain tensor and in general case all three components of the rotation tensor from the analysis of rocking curves is for the first time demonstrated and least-square procedure of calculation provided the high steadiness of solutions with respect to the errors of experimental data is proposed. The useful relations between the components of the strain tensor and the changes of crystallographic parameters are also derived. The cases of coherent, partially or fully relaxed layer structure are considered and simple relations that can be used to identify the coherency degree of epitaxial layers are presented. The formulae given for the general triclinic crystal are described in detail for most widely used cubic and hexagonal crystal system. In addition in our formulae the second-order approximation terms are also taken into account and this allows one to use the method proposed to evaluate highly mismatched material systems.

The strained state in ion exchanged H:$LiNbO_3$, H:$LiTaO_3$ and Me:$LiTaO_3$ structures widely used as high-quality optical waveguides is determined. The method of independent determining both the lattice parameters of unstrained hexagonal (trigonal) solid solution whose composition is equal to that on the surface of corresponding strained layer is for the first time proposed. Using the measured deformations and calculated crystal lattice parameters the number of distinct phase are identified in $HNbO_3$–$LiNbO_3$ $HTaO_3$–$LiTaO_3$ systems and phase diagrams of these systems are first plotted. The appearing exchange-induced mechanical stresses, the changes of refractive indices due to photoelastic effect and the compositions of formed structures are calculated.

Relaxation Process and Lattice Perfection of Buffer Systems for a HgSe Layer Investigated by High Resolution X-ray Diffraction

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We report on the investigation of different kinds of epitaxial buffer systems for a HgSe layer grown by *M*olecular *B*eam *E*pitaxy (*MBE*).

The ordinary buffer for HgSe is a thick ZnTe layer ($\geq 2\mu$m, $\Delta a/a \approx +0,31\%$) on a bulk GaAs substrate [1]. Despite the considerably large thickness necessary to reduce the dislocations generated by the large mismatch between ZnTe and GaAs ($\Delta a/a \approx -7,95\%$) a residual strain cannot be avoided. The consequence is a considerably long time for the MBE-growth and still a limited crystal perfection of the final HgSe layer on top. An alternative substrate with the advantage of a extremly low misfit with respect to ZnTe is GaSb ($\Delta a/a \approx -0.12\%$), so that a smaller thickness of the ZnTe is sufficient. The change in the sign of the misfits between HgSe/ZnTe and ZnTe/(bulk substrate) will be explicitely considered.

An ideal HgSe-matched buffer is realized by use of the ternary layer $ZnTe_{1-x}Se_x$ with x $\approx$ 0.05. We investigated the mentioned systems for different thicknesses of all layers in question with respect to relaxation state and crystaline perfection, using a (+n,-n,+m)-crystal-arrangement or a Bartels-monochromator for the measurement of symmetric and asymmetric reflections. We determine the ZnSe content by high resolution x-ray diffraction via Vegard's law.

In spite of a fully matched HgSe-buffer using a ternary layer we used an alternative method, having proved its effiency for III-V compounds and to our knowledgement applied for the first time to II-VI materials. To bend off the dislocation lines parallel to the interface buffer/HgSe a sequence of superlattices with different periods consisting of $ZnTe_{1-x}Se_x$ and ZnTe with a final cap fully matched to the HgSe lattice constant of high cristalline perfection near the perfection of the substrate is inserted in the buffer.

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STUDY OF $Si_{1-x}Ge_x$ BULK CRYSTALS BY MEANS OF HIGH-RESOLUTION DIFFRACTION

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Bulk crystals of $Si_{1-x}Ge_x$ solid solutions as an alternative for layer structures are important materials for faster semiconductor devices. The main problem is the growth of the solid-solution crystals with suitable structural perfection and chemical homogeneity. In order to characterize especially the inhomogeneity of $Si_{1-x}Ge_x$ crystals grown by float zone-technique, slices with $x = 0.02$ to 0.05 have been studied by means of double-crystal topography and triple-crystal diffractometry . It is the general aim of this study to contribute to a better understanding of the features of the defect structure in solid-solution crystals.

The $Si_{1-x}Ge_x$ crystals show distinct growth striations, predominantly with rotational symmetry around the the growth axis. In most cases, reciprocal space maps of these crystals are not isometric. The longer axes of the distributions are inclined to the diffraction vector. The inclination angle depends on the reflection as well as on the local position on the specimen surface. These results can be explained using a simple model applied by us already to another bulk solid-solution system [1]. The "striation planes" can be regarded as locally flat so that the relationships for diffraction angle and orientation changes as given for heteroepitaxial layer systems [2] should be valid. According to this model, for an otherwise perfect solid-solution crystal with fluctuations of the chemical composition it results a rod-like intensity distribution in the reciprocal space inclined about a certain angle. Assuming full lattice matching parallel to the striation planes, the inclination of these planes to the surface can be derived. On the other hand, if this inclination is known, e.g. from topographs, the degree of relaxation, if any, can be calculated.

The investigated specimens show more or less distinct chemical inhomogeneities, which can be interpreted by means of this simple model. Partial relaxation was found near the edges of the slices. The halfwidths of the distributions of relative lattice parameters integrated perpendicular to the diffraction vector are in the order of magnitude 5×10^{-5} corresponding to concentrations of 0.1 at%. Broadening of the rod-like distibutions is due to non-correlated either lattice-parameter or orientation distributions (or both) connected with the general defect structure of the crystals.

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HIGH RESOLUTION X-RAY DIFFRACTION INVESTIGATION OF CRYSTAL PERFECTION AND RELAXATION OF INGAAS-QUANTUM WELLS

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InGaAs-strained quantum wells (QWs) are widely used as active layer for high power semiconductor lasers. High output powers of more than 1 W and device lifetimes of more than 10^4 h require highly perfect material. For laser wavelengths in the region of 1017 nm a high In-concentration in the QW is needed. In that case the thickness of the QW reaches the critical one, where the relaxation process starts.
High resolution X-ray diffraction (HRXRD) is applied to study the crystal perfection of InGaAs layers, grown by metalorganic vapour phase epitaxy at different growth conditions. InGaAs-QWs sandwiched by a 100 nm thick GaAs cap layer and a 300 nm thick GaAs buffer layer were grown on a (001)-GaAs substrate. The thickness of the QW was varied from 15 to 25 nm at a constant In concentration (x = 0.16) to investigate the onset of relaxation and the defect formation at growth temperatures between 600°C and 750°C.
Keeping the QW-thickness constant the effect of increasing In-partial pressure in the vapour phase on In-incorporation into the QW was studied.
For the determination of the QW-thickness t and the In-concentration x (004)-$\Omega/2\Theta$-rocking curves were recorded and compared to simulations. For the samples grown at 650°C and 700°C the rocking curves can be simulated with sharp well profiles, whereas the samples grown at 600°C and 750°C show deviations from a sharp profile. The reasons for that behaviour will be discussed.
Area maps were taken around the (004), (115) and (-2-24) reflections to measure the QW-lattice parameters $a_\perp$ and $a_\parallel$. That way the degree of relaxation was obtained. From the spreading of the layer peak in the reciprocal space, the tilt of the QW lattice planes with respect to the substrate due to the misfit dislocation formation and the onset of 3-dimensional growth was determined.
The results for thin InGaAs-layers are compared to those obtained for thick, relaxed InGaAs-layers. Differences in the relaxation behaviour depending on growth temperature and In-content are deduced from the interpretation of area maps.
Cathodoluminescence (CL) was used to study the type of defects involved in the relaxation process. The results of HRXRD and CL were compared to those obtained by photoluminescence and transmission electron microscopy. This comparison leads to a detailed picture about the defect formation process in strained InGaAs layers.
For growth temperatures $\geq$ 650°C at $x \leq 0.25$ the layer-by-layer growth takes place. The relaxation of the lattice with increasing layer thickness is proceeding in the classical way by misfit dislocation formation. For T = 600°C and higher x-values no misfit dislocations are observed by CL, but in the HRXRD-area maps the layer peak shows a large spreading. This can be explained by a large mosaicity of the layer due to a 3-dimensional growth.

X-RAY DIFFRACTION STUDY OF NANOSTRUCTURES: THE OPTIMUM CHOICE OF DIFFRACTOMETER SAMPLING VOLUME

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X-ray determination of structural parameters of compositions with nanometric scale elements is a challenging problem. In these structures, the scattering intensity involves at least four contributions which are: (a) a structure size effect; (b) the size and shape irregularities and disturbances; (c) defects of crystal lattice; (d) compositional changes. Besides, their intensity can hardly be adequately quantifiable, since the relation between the extinction length and coherent scattering areas in different directions might considerably differ; in such cases, the feasibility of either a dynamical or kinematical approach is undetermined. At last, the X-ray observation of nanostructures is substantially impeded by the weakness of the scattered intensity. The solution of these problems depends on appropriate choice of diffractometer sampling volume.

In this communication, triple crystal diffractometer was applied to study: (i) relaxed superlattices; (ii) quantum wires and (iii) quantum dots. In order to develop an optimum sampling area all means available were utilized, such as a divergence (angle as well as spectrum), a diffraction geometry, a wavelength, a scan mode. Weak signals were detected by the use of schemes permitting the subtraction of the matrix background (with an analyzer in Laue geometry).

It was demonstrated that the diffracted intensity can be considerably saved when a probe affords "directional" resolution: in reciprocal space, the latter is kept high along the track, while in other directions intensity is integrated. With changing the track, a researcher covers the whole distribution, and the obtained data are sufficiently high resolved. The simulation of mismatched relaxed superlattices allowed to characterise misfit dislocation networks (a presence, a localisation, a density). Diffuse scattering were shown to be detected and analysed with a Laue geometry third crystal. The study of quantum dots with small-angle scattering was attempted.

The established principles allowed to design the optimum diffractometer sampling volume for different types of nanosructures.

X-RAY TOPOGRAPHIC STUDY OF DIRECTLY BONDED SILICON WAFERS WITH GROOVED INTERFACES

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The paper deals with Si structures fabricated with a technique whose ability to create bonds between atoms in joined materials has recently attracted much attention. The technique is known as direct bonding; it implies an intimate contact between appropriately treated mirror polished surfaces followed by heating. In our case, the surfaces were specially hydrophilisated, and annealing was made in the air. The modification of the technique was of such sort that the surface of one wafer was photolithography prepared as an orthogonal net of grooves. Initially, the fabrication of highly doped stripes was aimed on the improvement of electronic parameters of some special types of high power devices. Experimentally, it was shown that, during heating, grooves provoked a rearrangement of dislocations in the interfacial region.

A basic means for the observation of wafer interior was X-ray diffraction topography (XRT). STEM and laser scan techniques were also applied. In order to stress the role of grooves, the comparison of bondability of slightly as well as strongly misoriented wafers was attempted. The violation of coincidence relationship led to the appearance of high dislocation density whose transformation with annealing was detectable with XRT. As usual, the final aim was to obtain continuously bonded structures with relatively low defect density. We found that this purpose was attainable with the assistance of grooves whose appropriately chosen shape and width permitted a considerable decrease of dislocation density over sufficiently large sample areas: at high temperature, grooves efficiently collected dislocations adjacent to the boundary between the bonded wafers. The theoretical evaluation of driving forces promoting the dislocation glide and climb showed their substantial excess over the flow stress value, and thus confirmed the feasibility of the observed processes. A model was proposed to explain a detected transformation of interfacial defect arrangement into the most energy favourable state. Besides, a void appearance was also studied. Here the voids occurred to be air traps in which the pressure of hot air provoked an intensive dislocation movement. It was established that the voids were flatten with annealing. Relevant calculations allowed to interpret the bond strengthening with heating as due to the "dissolution" of the cavities under the compressive stress accompanied by vacancy diffusion and dislocation escape trough the free surfaces.

APPLICATION OF X-RAY DIFFRACTION IN LAUE GEOMETRY TO IMPERFECT NEAR SURFACE LAYERS

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Diffraction reflection geometry is known as the tool for observation of various types of damage below a crystal surface. Strain, roughness, composition etc. have been intensively studied in two as well three crystal schemes. At the same time, a complexity of intensity distribution has still demands approaches which permit a separate extraction of components. For this purpose, the change of diffraction geometry seems promising. The application of transmission setting to many structures is limited with high absorption and/or damage of crystal matrix (a substrate, a crystal bulk etc.).

For this communication we chose the structures whose absorption was relatively small and whose matrix contribution was negligible: MBE GaAs/Si compositions, VPE grown SiGe/Si HS's and boron diffusion layers in Si crystals. In order to assess their quality, a three crystal diffractometer was used.

It was demonstrated that the Laue geometry allowed to determine the following structural parameters:

– strain components;
– size of coherently scattering areas;
– compositional changes.

Strain components involved: (a) in-plane lattice spacing; (b) residual stress in partially relaxed HS's; (c) tetragonal distortion (for (001) and (110) oriented cubic layers); (d) microdistortion tensor (evaluated together with the Bragg geometry measurements). The in-plane data allowed to detect misfit dislocation networks and to evaluate their density. The in-plane spacing depth profile showed the distribution of the networks across the depth. In structures with high defect densities, a dislocation arrangement was interpretable due to microdistortion tensor values.

In Bragg case, a size contribution is known to be mixed with that of strain and easier revealable at smaller angles. The transmission setting also provides an opportunity to measure a size component. Besides, in many strongly damaged structures the in-plane and out-of-plane size might differ.

The compositional changes were detectable not due to strain variation, but due to redistribution of electron density.

HIGH RESOLUTION IMAGING OF ELECTRONIC DEVICES USING LINE MODIFIED - ASYMMETRIC CRYSTAL TOPOGRAPHY (LM-ACT)

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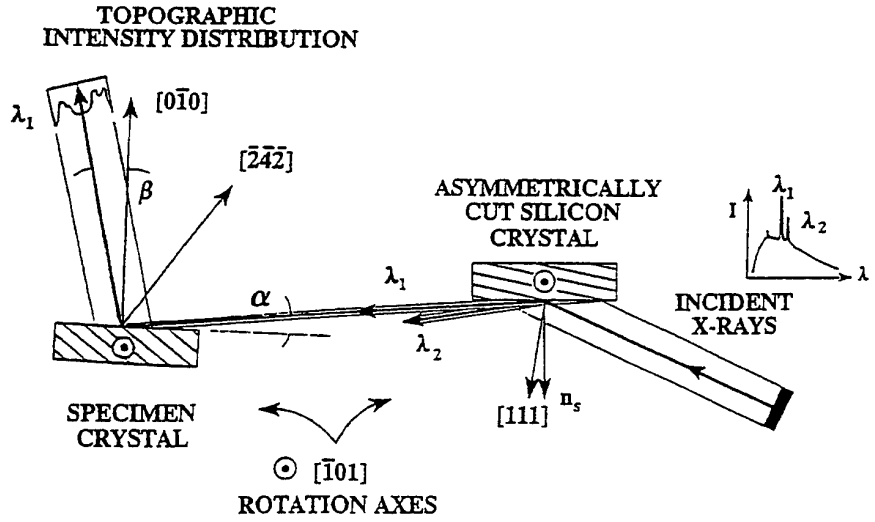
ABSTRACT

X-ray topographic measurements are reported for implantation and superstructure details in an otherwise perfect, parametric, single crystal device. Implantation or deposition strains, x-ray absorption, and surface shadowing or enhancement of the x-ray beam contribute to the observed contrast. Sequential topographs have been obtained to monitor the stages of device fabrication [1]. Stereo-pair images have been obtained and superposed to provide a depth perspective. Comparison is made with dynamical theory calculations. The x-ray penetration depth and micrometer size of the thin film nuclear emulsions used to record the diffraction images are shown to limit spatial resolution [2].



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STUDY OF DEPTH DIFFUSION PROFILES OF Zn IN InP BY HIGH RESOLUTION X RAY DIFFRACTION AND X-RAY STANDING-WAVES TECHNIQUES

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The diffusion of Zn into the III-V semiconductors is a very important technological process. Zn atoms occupy indeed group III sites, behaving as acceptors and, thus, producing p-type electrical properties. A good deal of research has been carried out on Zn-diffusion into III-V crystals and it is generally accepted that Zn atoms diffuse by a substitutional-interstitial mechanism: As a peculiar characteristic of InP, a large discrepancy between the hole concentration and total Zn concentration has been reported by many authors, the former being one order of magnitude lower than the latter.

An interesting problem concerns the lattice strain produced by Zn diffusion into InP crystals. Since it may be expected that a large concentration of Zn atoms do not occupy substitutional sites, it is quite difficult to theoretically predict sign and amount of the lattice strain. Moreover, no experimental results have been reported to date. For this reason an experimental investigation has been performed on Zn-diffused InP layers using high resolution X-ray diffraction (HRXRD) and standing wave (SW) method recording the X-ray photoelectrons (XRP).

Zn-diffused InP layers have been prepared in a sealed quartz ampoule at 500°C using both Zn_3P_2 and Zn+InP sources. The diffusion time ranged from 10 up to 90 min. As substrates, nearly dislocation-free S- and Zn-doped InP crystals have been used.

X-ray experiments were carried out by means of a computer controlled diffractometer using the Cu Kα radiation and the 004 reflection. The incident beam was conditioned by an U-shaped (004) Si monochromator. The SW experiments were performed in a conventional (n,-n) double crystal geometry using the above mentioned monochromator. in order to measure the XRP emission, the sample was mounted inside a gas-flow proportional counter and the electrons were recorded by a gold-plated tungsten wire parallel to the sample surface. A gas mixture (90% He+10% CH_4) flowing through the counter provided a collection efficiency around 100%. The energy resolution was 17% (at 8 keV) and the energy range was selected using an amplitude descriminator.

The results show that in the S-doped crystal the diffused/virgin interface is very sharp and the diffused layers are lattice contracted. The maximum strain close to the surface does not depend on the diffusion time, whereas the thickness of the diffused layer increases by increasing the time in a monotonic way. Both surface strain and diffusion lenght depend on the Zn source. Finally, no diffusion has been observed in the Zn-doped crystals

LATTICE DILATION IN Si- AND Sn-DOPED InP HOMOEPITAXIAL LAYERS

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Incorporation of doping impurities is known to produce lattice constant modifications in semiconducting crystals. As for InP, the effects of Ge, Zn, S, Sn have been studied in bulk crystals using the X-ray diffraction Bond method, that permits to measure with high accuracy the lattice constant on absolute scale. Moreover, the effects of S and Te have been investigated in InP homoepitaxial layers using double crystal diffractometry (DCD), which provides accurate values of the difference between the lattice constant of the doped layer and that of the undoped substrate.

The present work concerns n-type Si- and Sn-doped InP homoepitaxial layers grown on (001) Fe-doped semi-insulating InP substrates by metal organic vapour phase epitaxy and liquid phase epitaxy respectively. The net carrier concentration N_d-N_a has been determined by Hall measurements, while the total dopant concentration N_T has been measured by secondary ion mass spectrometry. In range investigated ($4x10^{16}$ cm^{-3} < N_T < $2x10^{18}$ cm^{-3}) the relations $N_T = 1.4x(N_d-N_a)$ and $N_T = 5.7x(N_d-N_a)$ have been found for Si- and Sn-doped layers respectively. Non-unity relationships can be due to either donor compensation by acceptors or incorporation of the impurities as electrically inactive interstitials or complex defects.

The lattice mismatch between layer and substrate has been carefully measured as a function of N_T by DCD using the Cu $K\alpha_1$ radiation, a nearly perfect InP first crystal and the 117 slightly asymmetric reflection which has as larger sensitivity to the mismatch than the conventional 004. A lattice dilation has been observed for both dopants, but Sn doping has been found to be about 8 times less effective than Si doping. The experimental findings cannot be explained by the Vegard's law, which predicts in fact a lattice contraction for both the dopant impurities. Therefore, the role of the free carriers in producing the lattice dilation can be reasonably suggested.

X-RAY TOPOGRAPHY AND HIGH-RESOLUTION DIFFRACTOMETRY OF BONDED SOI FILMS

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Bonded silicon on insulator (BSOI) materials provide significant new opportunities for high-performance microelectronic devices. From the point of view of X-ray diffraction these materials present some distinctive structural characteristics. Using double crystal X-ray diffractometry (DCXRD), the individual reflections from the bonded layer and its underlying substrate can easily be separated due to misorientation between the two original wafers. Comparing the rocking curves with dynamical simulations and accounting for wafer bending and layer thickness, the layer peaks are still significantly broadened by at least a few arcseconds. A characteristic microstructure in these layers is also revealed by X-ray topography. In our laboratories, X-ray rocking curves and single crystal topographs have recently been obtained from a series of wafers with 2 μm bonded layers on 1μm buried oxide. Results were found to be most strongly dependent on the surface roughness of the starting wafers. These results are presented in this paper together with further results from thinner bonded layers (down to 0.3μm). Triple crystal diffractometry and double crystal topography results are also included . This work illustrates the value of applying a range of advanced X-ray techniques to the research of important structural properties in a technologically exciting family of novel materials.

X-ray topography, TEM and IR studies of three-step annealed Cz Si

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Czochralski grown silicon single crystals of different oxygen content [1] and *n* or *p* type, annealed at temperatures of 750°C (4 or 5 hours), 1050°C (1 h) and 1150°C (4 hrs) were investigated by X-ray Lang and section topography as well as transmission electron microscopy. The X-ray topographic studies were performed using $MoK_{\alpha 1}$ radiation and symmetric reflections (440, 660 for [001] oriented samples and 440, 660, 224 and 448 reflections for [111] oriented samples) as well as asymmetric ones (333, 335 for [001] samples and 333, 335, 004 and 117 for [111] samples).

Lang topography revealed diffraction contrasts for spherical defects with high density for the *n*-type [001] oriented samples. On the contrary, for the *p*-type [001] or [111] oriented samples only few such defects were observed. Differences in the visibility of the Kato fringes in section topographs taken with asymmetric and symmetric reflections suggest the presence of an asymmetric long-range deformation of these crystals. Comparison of the results for the same crystals annealed in two-step and three-step processes [2,3] suggests a difference in the formation of the oxygen-related defects and overall perfectness for the *n* and *p* type samples. By means of TEM various kinds of defects were observed [4]; for instance, spherical precipitates and their agglomerations, dislocation loops and dipoles with different dimensions (in the range of 0.1-1μm) and densities.

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X-RAY DIAGNOSIS OF LASER DIFFISION OF ALLUMINIUM INTO SILICON

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Diffusion processes occur at almost every stage of semicondacting materials technique. Structural changes caused by termal diffusion have been described on the basis of X-ray diffractometry data. Structural changes caused by laser diffusion have been studied less. This work is aimed at diagnosing the near-surface structure changes caused by laser diffusion of alluminium into silicon.

The investigation of the structure was conducted by double- and triple-crystal X-ray diffractometers in non-disperse geometry (n,-n) and (n,-n,n) accordingly.

Perfect Si crystals with reflection (111) functioned both as a monochromator and analyser, Cu Kα -radiation being the X-ray sourse.

The samples under study were prepared in the way described below. The plane (111) of Si monocristals was coated Al layer 960 ± 80 Å thick. The coating was carried out in vacuum $P= 1{,}33*10^{-2}$ Pa. The planes of the samples opposite to those coated by sprayed alluminium were irradiated with CO_2 laser. The time of the irradiation varied from 1 to 3 sec. The impulse capasity density was 30 kwt/cm

The crystal lattice defomation profile in the near-surface layer, the type of defects and the optimal duration of the laser impulse were determined by laser diffraction. The results obtained show the cardinal difference between laser diffusion and the equivalent thermal one. The laser diffusion layer is thicker by the order of 2 than the thermal diffusion layer, the suface heating temperature and the time of the irradiation being the same for both techniques. All distribution has a more uniform profile.

X-RAY TOPOGRAPHIC CONTRAST FROM ION IMPLANTATION REGIONS IN SILICON CRYSTALS

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A systematic experimental study of X-Ray topographic contrast has been performed on model Si crystals after **Tl** and **P** ions implantation through a photo gauge. The ion irradiated regions had a form of parallel lines 30 μ wide crossed all the surface of samples. The complex of X-Ray topography methods has been used for studies: the Borrmann technique, the Lang method for section and projection topography, the Berg-Barrett method, a reflection section topography and section topography in grazing diffraction geometry.

The boundaries of irradiated regions give contrast on projection topographs of all samples. The contrast looks as dark zones of different intensity in the case of "thin" crystal and it is of alternate opposite sign at neighbour boundaries in the case of "thick" crystal. It gives possibility to determine the sign of the deformation gradient at the both sides of boundary with use only one projection topograph and unambiguously identify the irradiated regions. A "vanishing" of contrast from boundaries occur if the diffraction planes are normal to the boundaries orientation.

A section topography in the grazing diffraction conditions shows a spectacular peculiar contrast which gives a possibility of visualization of regions with amorphized surface layer apart from the boundary images in the cases when the implantation doze is sufficient for amorphization.

Section reflection topographs show complicated periodic contrast from boundaries and irradiated regions. Mode of contrast depends on the mutual orientation of boundaries of irradiated region to the dispersion plane. If they are parallel the series of successive contours can be observed. The geometry of countors depends on iimplantation doze and a kind of bombarding ions. An additional periodic contrast not related directly with periodicity of irradiated regions can be observed when the boundaries arranged at some angles to the dispersion plane.

Transmission section topographs show deformations of characteristic interferention fringes of perfect crystal. The picture of distortions is also sensitive to irradiation doze and kind of bombarding ions.

Details of distribution of interferention fringes on the transmission section topographs can be a good basis for the calculations of normal and tangential components of real elastic stresses formed as a result of ion implantation.

SECTION TOPOGRAPHS UNDER DC ELECTRIC FIELD OF HYDROGEN IMPLANTED $LiNbO_3$

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Lithium niobate ($LiNbO_3$) displays a number of large-magnitude physical effects which make it an attractive material for applications such as guide-wave optics. For example, ion implantation of $LiNbO_3$ is a method for writing optical circuits for use in integrated optics [1]. Implantation by energetic (~ MeV) light ions such as hydrogen or helium has the effect of considerably lowering the refractive index in the implanted region, while the refractive index of the layer only traversed by the implanted ions generally remains approximately the same as the bulk one [2-4]. The region of lower refractive index (implanted layer, thickness between 0,2-1 μm) can thus define the boundary of a waveguiding area (traversed layer, thickness up to 15 μm). By varying the ion dose and energy a variety of waveguide profiles can be generated.

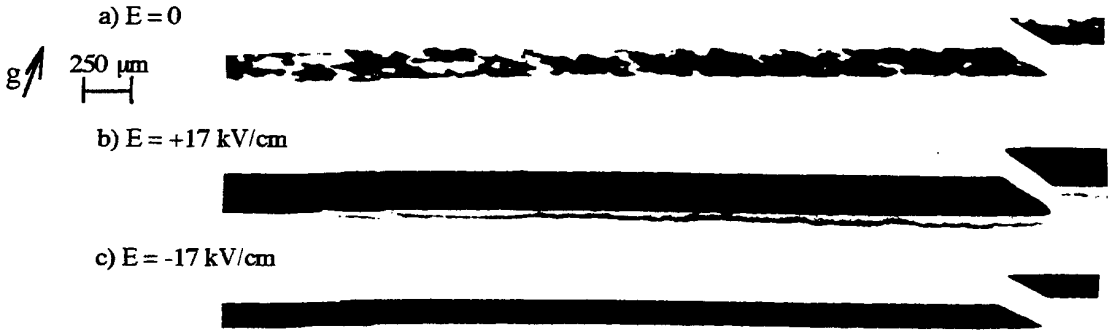

Fig.1 : Section topographs of $(20\bar{1}6)$ reflection, $\mu t = 1.2$ (μ is absorption coefficient and t thickness of the sample), $\lambda = 0.226$ Å, g is projection of diffraction vector on the film

The aim of the present work is, by using section topographs, to characterize traversed and implanted layers of $LiNbO_3$ (and α-$LiIO_3$) crystals implanted by hydrogen. Fig.1 shows section topographs of a $LiNbO_3$ crystal, platelet-shaped, with the main surfaces perpendicular to the Y axis, without field and under field applied along the Y axis. The image of the layer associated with the implantation moves either inside or outside the image of the bulk when changing the polarity of the applied field. The experiments indicated that the implanted layer is single-crystalline, slightly misoriented with respect to the bulk and is observable without a field. Under a field, it is invisible. However, the layer traversed by the implanted ions when observed under a field gives rise to a line parallel to the surface which is more misoriented with respect to the bulk than the implanted layer without a field. The misorientation of the image of this traversed zone is proportional to the field and seems to be strongly linked with the piezoelectric effect.

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DEPTH DETERMINATION OF AMORPHOUS–CRYSTAL INTERFACES OF BURIED AMORPHOUS LAYERS IN IMPLANTED SILICON BY X–RAY DIFFRACTION, RUTHERFORD BACK–SCATTERING AND TRANSMISSION ELECTRON MICROSCOPY

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High–resolution double–crystal X–ray diffraction was used to investigate the depth distribution of lattice damage produced in silicon by ion implantation. Silicon and arsenic were used as the incident ions at 1.5 MeV and 0.8 MeV, respectively. For sufficiently high doses, the amorphization threshold is exceeded and a continuous buried amorphous layer is formed. Best–fitting of the experimental rocking curves was made by dynamical diffraction model and automatic minimization routines. The information of this procedure are in terms of the depth profiles of lattice strain and static atomic displacement. The latter parameter was found to increase sharply in the central region of the strain distribution and its very large values. as directly obtained by the minimization routines, demonstrate undoubtely that amorphization occurred in this embedded region. The front and back amorphous–crystal (a–c) interfaces can then be easily located on the depth scale.

The samples were subsequently heated at 400 °C temperature for 10 min in order to make the interfaces flatter and both X–ray measurements and rocking curve best–fits were repeated. The a–c interfaces were found to move slightly towards one another indicating beginning of recrystallization.

Rutherford back–scattering in the channelling configuration and cross section transmission electron microscopy were used to compare the depth values of the a–c interfaces before and after annealing with those resulting from rocking curve analysis. The correspondence was found rather good if the different sensitivities to damage levels and the errors of the three techniques are taken into account. The influence of the interface roughnesses is also discussed.

The comparison indicates that high–resolution X-ray diffraction is a technique able to study also buried amorphous layer and that the often neglected static Debye–Waller factor is a valuable parameter for this study.

TWO CRYSTAL FORMS OF AN RNA PSEUDOKNOT

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The RNA pseudoknot is a very important tertiary motif widely found in virtually every class of RNA. In this motif, nucleotides from the loop region of a hairpin pair with a single stranded region outside the hairpin to form a quasi-continuous helix with loops crossing the major and minor groove. Recent evidence shows that pseudoknots are potent inhibitors of the reverse transcriptase of HIV, the virus that causes AIDS. To date, no detailed information of the geometry of this motif from a crystal structure is available.

We were able to produce highly ordered single crystals of an RNA pseudoknot from Beet Western Yellow virus. This pseudoknot fragment is 28 nucleotides in length and involved in translational frameshifting. Crystals of two different forms with diverse characteristics were obtained One crystal lattice is trigonal, space group P3(1)21, unit cell a=30.84, b=30.84, c=144.55, α=90.0, β=90.0, γ=120.0, with 1 molecule in the assymetric unit. These crystals are tightly packed with a low content of solvent. Despite of the small size (0.05x0.06x0.2), they diffract to 1.9 Å. Flash freezing to 100K are essential for stabilizing the crystals to maximum resolution. These small yet highly ordered crystals are most suitable for synchrotron data collection. The other crystal form is cubic, I23, a=b=c=108.9. Crystals are chunky (0.5x0.5x0.5) but fragile with a high solvent content, the diffraction limit is 2.8 Å. Structure analysis will proceed with Multiple Isomorphous Replacement and Molecular Replacement methods. The comparison of the pseudoknot structure in the two crystal forms will allow us to gain sufficient knowledge of this motif. In addition, these studies will have implications on how lattice packing and solvent content can effect the diffraction of crystals.

X-RAY TOPOGRAPHY OF COMPLICATED CROSS-SECTION SAPPHIRE SHAPED CRYSTALS

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Formation of real structure of sapphire crystals of the shapes shown on the figure was studied. Definition the mechanism of mosaic structure origin absolutely important for management by structure and properties of crystals was the general aim of the work.

X-ray topography and X-ray analysis methods were used. Application of one and two-crystal arrangements for back diffraction and television camera allowed us to investigate the crystals with natural surfaces as well as samples of big sizes and of curved shape.

It was shown that initial part of crystals shaped as angle bars corresponding to the seading region and growth up to steady-state has special significance for real structure formation. Mosaic structure is born in the same stripe parallel to growth axis under seading region and is determined by thermal stresses. The subsequent development of structure takes place under action thermal stresses and its level increasing up to steady state transition. The structure of smaller face is always better than bigger one. There is the orientation dependence of crystal perfectness for not only growth axis but for two faces also.

The mosaic structure of crystals shaped as tubes also depends on seed shape. We have discovered the rotation of crystals-tubes around growth axis on an angle up to some degrees, which is the crystal accommodation reaction to growing conditions and determines the bloks structure character.

Analysis of our results allowed us to propose some manners for management by real structure formation both on initial and steady-state growing stadies. As the result the crystals with various complicated cross-section and better structure available for technical using were grown.

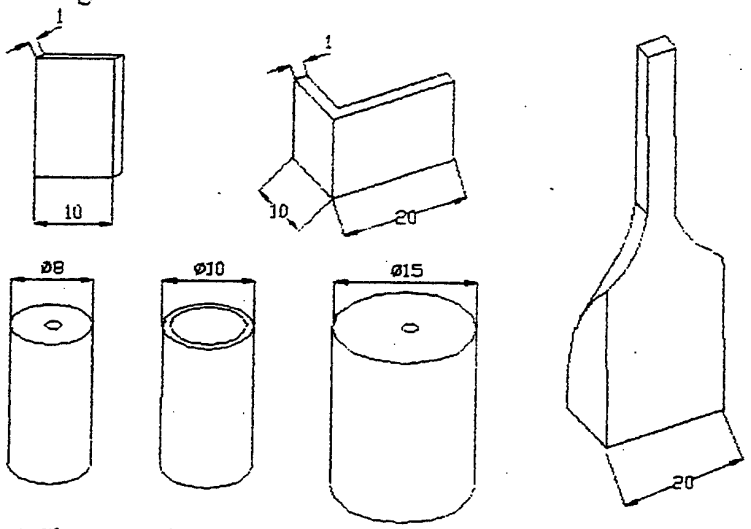

Fig.1 Shapes and dimensions of crystals wich have been studied.

STRUCTURAL DISTORTION ANALYSES OF BORON-DOPED SILICON MONOCRYSTALS BY THE RESULRS OF TRIPLE-CRYSTAL X-RAY DIFFRACTOMETRY

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Triple-crystal X-ray diffractometry is efficient technique for structure stude of the distorted perfect crystal subsurface layers. This method was applied to the stude of Si monocrysrals (plane (111)) doped with boron ions with E=25 kev and dose $D=1.875*10^{15}$ sm^{-2}. The samples doped were subjected to isothermal annealing under nitrogen at temperature range from 300° to 1000°C, the time range being from 10 to 120 min.

The stude was conducted on the triple-crystal X-ray diffractometer (TRD) in non-disperse geometry (n,-n,n) according to CuKα-radiation scheme. Nearly-perfect silicon monocrystals were employed as an analyser and a monochromator.

The ion-doping resulted in a positive subsurface layer deformation with maximum value $\Delta d/d \approx 0.003$ at about 0.005 mkm of depth from the surface. The annealing at the temperature range from 300° to 600°C didn"t lead to a notable change in the outer layer structure. The analyses of the TRD spectrum intensity presented at the annealing temperature of 800°C and the time of 10 min testify to the near-surface formation of two layers with negative and positive deformation parameters. The layer with positive deformation diposed near to the surface. It is noteworthy that at temperature range from 300° to 800°C the diffuse peak on the TRD spectrum was not present. The annealing temperature increase to 900° and 1000°C resulted in the vanishing of the positive deformed layer. The negative deformatiom calculation are found to decrease with the increase of annealing temperature and the time, the presence of intensive diffuse peak being also characteristic. The analyses of the dependence of the diffuse peak intensity on the sample deflection angle show the chaotically distributed dislocation (1000°,60 min) and the defects of dislocation loops type (1000°, 120 min) to contribute greatly to the diffuse peak.

SYNCHROTRON X-RAY TOPOGRAPHIC STUDY OF STRAIN IN SILICON WAFERS WITH INTEGRATED CIRCUITS

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The objective of the study is to determine the strain by means of X-ray diffraction topography in silicon wafers which have gone through a process for integrated circuits employed in mobile phones and tv monitors. The topographic observations are supplemented by electrical measurements such as the threshold voltage of a transistor in order to find the influence of the strain on the device performance and yield.

Section topographs made with synchrotron radiation at HASYLAB show the strain field below the surface as a series of curved lines starting at one edge of an oxide layer and ending at its other edge. The topmost passivation layer on the wafer surface adds to some strain, which gives rise to increased intensity. In addition the picture shows a few oxygen-induced large defects and a lot of small ones. A large-area topograph taken from the same wafer shows clearly the outlines of the contact pads as well as the very small dot-like defect images. The deepest strain field measured from the surface is caused by the pads of a chip. The contact metallisation of the devices shows up as streaks in the section topographs.

CHARACTERIZATION OF SiGe HBT-STRUCTURES BY DOUBLE AND TRIPLE-CRYSTAL DIFFRACTOMETRY

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SiGe heterobipolar transistors (HBT) offer in comparison with the conventional Si transistors the possibility of significantly increased features (high transit frequencies, low noise etc.) and the advantage of a simple integration into the well-established Si device technology. Maximum oscillation frequencies of 120 GHz have been demonstrated. This means, that SiGe HBTs are in principle able to compete with III-V semiconductor devices.
Besides other constructive factors, the vertical profile of the germanium content as well as the one of doping atoms is decisive for the parameters to be achieved. This vertical structure is produced by an epitactical deposition, where molecular beam epitaxy (MBE) and chemical vapour deposition (CVD) techniques are the prefered methods. Diagnostical analysis techniques are important to prove, whether the given parameters are actually obtained.
The x-ray diffractometry is a powerful tool to determine the Ge profile, since the lattice constant of a $Si_{1-x}Ge_x$ alloy is depending on the Ge content x in a rough linear approximation according to $a_{SiGe} = a_{Si} (1 + 0.0418 x)$. This method is nondestructive and allows the characterization of the layer before the final device preparation is done or after this preparation as far as a sufficiently large nonstructured area exists on the wafer.
The typical Ge depth profiles of an HBT structure consists of a SiGe layer 25...50 nm thick burried under a some 10 nm thick Si cap layer, where the maximum Ge content of 20...25% is constant on a plateau followed by a linear decrease in the direction to the surface. The influence of additional dopants on the lattice constant is of minor importance.
Double- and triple-crystal diffractometer (DCD and TCD) arrangements were used to measure the rocking curve (CuK_α-radiation, 400-reflection) of such HBT structures. Even the individual layers (Si-cap, SiGe layer) are relatively thin, a characteristic interference pattern can be observed that offers the possibility to determine the deformation profile and following the Ge depth profile by fitting a calculated rocking curve (RC) to the experimental one.
Both diffractometer techniques will be compared. The high resolution TCD in parallel (n,-n,n) arrangement using the main peak analysis method provides more detailed RCs due to the reduced influence of diffuse scattering. On the other hand, it requires much more measuring time. Additionally, TCD allows a proof of the structural perfection (pseudomorphic growth) of the SiGe layer by the analysis of diffuse scattering of possible misfit dislocations.
It will be shown that this measurement allows the determination of the Ge depth profile with an accuracy in the order of 1 nm. Small changes of the profile parameters can significantly change details of the rocking curve. The possibilities to analyze additionally the boron doping profile within the Ge layer will be discussed. The results are compared with SIMS measurements.

INVESTIGATION OF STRUCTURAL DISTORTIONS INDUCED IN SEMICONDUCTOR CRYSTALS UNDER THE EXTERNAL INFLUENCES (RADIATION) BY MEANS OF X-RAY DIFFRACTION MOIRE METHOD

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Experimental investigations of structural distortions induced in silicon crystals by different doses of hard X-rays, γ-radiation, as well as 4.5 MeV electrons are carried out by means of X-ray diffraction moire technique.

A detailed review of the background of actuality of this topic (X-ray interferometric investigation of semiconductor crystals' distortions induced by radiation), is well known. Convinced that the irradiation of the crystals by hard X-rays, γ-quanta and high energy electrons cause the formation of point defects, one comes to a conclusion that investigations in this scope should be implemented by means of X-ray interferometry. Proceeding from the obtained topograms, we elaborated X-ray interferometric photopower method for experimental investigations of moire patterns' period dependence on radiation dose, as well as on the quanta and electron energy. A technique is elaborated and the point defects' density is determined in dependence on radiation dose and depth of radiation penetration. It is shown that the density of point defects is increasing with the increase of radiation dose, and decreases with the increase of electrons' penetration depth. It is also shown that the general influence during the irradiation of the crystal of the interferometer by hard X-rays is more than in case of γ-radiation because of smallness of absorption coefficient. X-rays' passage through the substance causes comparatively slight distortions.

POSSIBILITIES AND LIMITATIONS OF THE ASYMPTOTIC BRAGG DIFFRACTION METHOD

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The asymptotic Bragg diffraction method /1,2/ (known also as the truncated Bragg rods method /3/) is a powerful tool in characterization of thin surface layers, interfaces and superlattices. The possibilities inherent to the method are very wide, the depth resolution less than 1 A can be reached. However the receiving full information about the perfection of layer structure from experimental curves appears to be very complicated problem. Main difficulties come from the fact that triple crystal rocking curves can not be in principal interpreted in unambiguous way quite a lot of equivalent solutions exist simultaneously /4/. These and other related to the method problems have been analyzed in detail.

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Transformation of X-ray Polarization under Grazing Incidence Diffraction on the Crystals with Amorphous Layer.

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The interest in detailed theoretical and experimental studying of the X-ray diffraction under grazing incidence conditions stems from the wide possibilities of such geometry in investigation of the crystal surface structure. The influence of surface amorphous layer on the reflection coefficients under grazing geometry was analyzed in a number of papers [1-2].

In the present paper the transformation of X-ray polarization under the grazing incidence diffraction on crystals with an amorphous surface film is studied. The dependencies of polarization state of reflected and diffracted waves on parameters of incident wave with an arbitrary polarization are found in two-wave approximation of the dynamical diffraction theory. Strong spatial anisotropy of a crystal close to the exact Bragg condition manifest itself in a steep change of polarization of scattered waves near the critical angles of reflection. The degree of circular polarization of the reflected waves and orientation angle of polarization ellipse strongly depend upon the parameters of amorphous film. Spraying an amorphous film of optimum thickness on the crystal surface permits to transform incident linearly polarized wave into circular one with the degree of circular polarization close to unity and vise verse. As an example, numerical calculations for the diffraction of CuK_{α} -radiation on (220) planes of a germanium single crystal with an amorphous layer of various thickness are presented. In the case when dielectric susceptibilities of a crystal and amorphous layer are the same maximal effect can be reached at the film thickness ~ 10 nm.

The polarization transformation can be used for determination of the amorphous layer thickness as well as for monitoring the X-ray beam polarization states.

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DETERMINATION OF THE STRUCTURAL PROPERTIES OF GE-δ-LAYERS BURIED IN SI(100) BY A COMBINATION OF X-RAY REFLECTIVITY, GRAZING INCIDENCE DIFFRACTION AND X-RAY STANDING WAVE MEASUREMENTS

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MBE-grown crystalline δ-layers attract increasing attention in semiconductor technology both as building blocks for 3D integrated circuits and as low dimensional electron systems. For a wide range of applications precise structural characterization is mandatory.

We present a combination of x-ray studies of Ge-δ-layers buried in a (100) Si single crystal grown by MBE at 500 °C. X-ray reflectivity measurements in the vicinity of the critical angle of total reflection render the caplayer thickness and the roughnesses of surface and interfaces from model calculations using the Nevot-Croce regime of roughness.

Exit angle resolved Grazing Incidence Diffraction measurements (GID) were performed at the beamline D4 of the HASYLAB Synchrotron radiation facility. By this special GID technique structural information is obtained as a function of depth. The Bragg intensity distribution along the exit angle shows an oscillatory structure. From model calculations using kinematic scattering theory a depth resolved determination of the lateral lattice parameter is obtained together with the caplayer thickness and the roughnesses, which are consistent with the reflectivity measurements. Moreover the miscut angle between the surface and the (100) lattice planes could also be extracted from the model fit and its value is in good agreement with the standard x-ray diffraction result.

Finally the analysis of the Ge fluorescence under excitation of X-ray Standing Waves using two different sets of lattice planes allows for an extremely precise determination of the atomic positions of Ge inside the δ-layer.

In conclusion the combination of these advanced x-ray methods provides a complete structural description of buried δ-layer systems. As a consequence decisive improvements of the MBE growth process for these and other layer structures in general is to be expected.

THE GRAZING INCIDENCE DIFFRACTION OF X-RAYS WITH WAVE FRONT AMPLITUDE PERIODICALLY VARYING ALONG THE CRYSTAL ANTRANCE SURFACE (SYMMETRICAL LAUE CASE)

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The modern technology based on use of thin-layered materials with layer thicknesses in nanometre ranges, e.g. in integrated circuits, X-ray mirrors etc. Interfacial regions extending over a few monolayers can strongly degrade device performance (see Foster et al.), especially as the thicknesses of performing elements become rather thin with further miniaturization of devices. If the device performing elements are deposited periodically on the substrate, such layer is like a diffraction grating (DG). The reflection (or transmission) function of DG may be used for the control and monitoring of device surface elements quality.
With that end in view the following scheme is investigated: reflected from DG X-ray wave field falls at grazing angles onto a single crystal (SC). the reflecting net planes of SC are normal to entrance surface (symmetrical Laue case). DG has the following reflection (or transmission) function:

$$g(y)=t(y)*u(y)$$

where t(y) is rectangle unit impulse function with length L same as for DG; u(y) is an arbitrary periodic normalized function with the period D; L=ND; N is an integer; $1<<KD$; K is the wave number of incident X-ray plane wave. The description of reflected from DG wave field is given by the quasi-optics approximation. The incident wave field may be presented by yhe plane wave with wave front amplitude periodically varying along the SC surface. The surface waves' origin conditions are also obtained for this problem.

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SYNCHROTRON RADIATION X-RAY MULTIPLE DIFFRACTION IN THE STUDY OF DOPED KDP

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In this work, the X-ray Multiple Diffraction technique using synchrotron radiation is applied to study the habit modification of KDP samples induced by incorporation of trivalent transition metals.

X-ray Multiple Diffraction arises when an incident beam simultaneously satisfies the Bragg law for more than one set of lattice planes within a crystal. A set of planes, which is adjusted to diffract the incident beam, is called the primary planes (h_p,k_p,ℓ_p). By rotating the sample around the primary reciprocal lattice vector, Renninger scanning method (RS), several secondary planes (h_s,k_s,ℓ_s) within the single crystal with arbitrary orientation also diffract. The intensity interaction among the primary and the several secondary reflections are established through the coupling reflection (h_p-h_s,k_p-k_s,ℓ_p-ℓ_s). When the phases associated with the interaction between the primary and secondary beams constructively interfere we get an enhancement of intensity (Umweganregung) or a reduction of intensity (Aufhellung) in the converse case. The high resolution multiple diffraction set-up developed at station 7.6 of the Daresbury Laboratory was used to enhance the high sensitivity of this technique to detect small symmetry changes in the perfect KDP crystal lattice caused by the impurities. A model obtained from the Molecular Modelling Package (CERIUS2) has suggested the habit modifiers of transition metals are in a tetra-hydrated state absorbed at the substitutional site of phosphate in KDP.

Renninger scans of pure and Mn doped KDP were carried out using the 400 primary reflection since the impurities were segregated in this growth sector. Precise scans with steps of 0.002° around the ϕ=0° symmetry mirror together with the behaviour of observed secondary reflections were necessary to determine the used wavelength, λ = (1.910 ± 0.002) Å. The analysis of pure and Mn-KDP Renninger scan clearly confirms the presence of impurities through the suppression in the doped scan of the huge peak asymmetry characteristic of perfect (FWHM <3 arcsec from 400 rocking curve) crystal lattices. The associated peak broadening cause almost total disappearance of some aufhellung dips in the doped scan. Effort is now being directed to explain the extra umweg peak (impurity contribution) measured close to ϕ=1.13° by improving the MULTX simulation program.

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DEVELOPMENT OF X-RAY POINT FOCUSING TECHNIQUES USING CRYSTALS WITH TWO-DIMENSIONALLY MODULATED SURFACES

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Predicated upon of the application of the recent wave-optics approach to x-ray imaging using bent crystals,[1] the oscillatory behavior of the diffraction amplitude was revealed, and it was found that the intensity distribution at the focus is determined by the convolution of Bragg and Fresnel amplitudes over the bent crystal surface. It was suggested that an artificially modulated structure, determined by the distribution of the diffraction amplitude can be fabricated on the curved crystal surface to narrow the width of the Bragg diffraction amplitude, and in turn, the focal spot size, without reducing the peak.[2] Obtaining a point focusing by using a cylindrically bent crystal[3] is also possible by applying a modulated structure, defined by the Fresnel diffraction amplitude distribution, on the crystal surface along the sagittal direction. Based upon the Fresnel diffraction amplitude distribution, it was also proposed that a flat crystal with elliptically shaped zone structure can perform point-to-point focusing.

A silicon flat crystal with elliptical zone structures was made by using the electron-beam lithography technique. In addition, various zone structures were considered for use with different crystal reflections and focusing parameters, which could be applied to synchrotron beam lines, laboratory x-ray tubes, and plasma x-ray sources. Improvement of the fabrication with e-beam technology is in process, and the experimental set-up is currently being implemented to use an x-ray tube with Ti Kα, and a silicon flat crystal with elliptical zone structures and (111) reflections. The experimental results are expected to be compared to the theoretical estimates. The realization of this technique of x-ray point focusing will contribute to the advancement in x-ray optical elements for the fields of x-ray micron-diffraction, imaging, and micro-fluorescence analysis.

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LATERALLY RESOLVING X–RAY DIFFRACTOMETRY UTILIZING X–RAY SENSITIVE CCD CAMERAS AND ITS APPLICATION TO A GeSi LAYER SYSTEM GROWN BY AREA SELECTIVE EPITAXY

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High resolution x–ray diffractometry is a widely used tool to investigate layer systems in order to determine structure parameters. However, this technique finds its limitation for samples with laterally inhomogeneous properties. To extend this method for applications demanding lateral resolution the sample can be scanned by a fine x–ray beam (several commercial HRXRD permit this mode) or a lateral resolving detector has to be used (a technique utilizing a one–dimensional position–sensitive detector has already been introduced [1]). A two–dimensional lateral resolving detector benefits by a fully parallel registration saving a scanning of the sample. For quantitative measurements x–ray sensitive slow–scan CCD cameras are used. With such kind of electronic detectors digitized data are immediately available for image or data processing purposes.
Two alternative concepts are possible: 1. Using a phosphor x–ray quanta are converted to visible light, which is amplified in an image intensifier and detected by a CCD camera. 2. X–ray quanta directly interact with the silicon material of the CCD sensor without any preceeeding conversion.
Both types of cameras have been used. For our purpose single x–ray photon counting and a resolution of at least 20 µm are required which can be fulfilled with both concepts.The imaging performance of both types of detectors will be demonstrated. Results and a first diffractometric application of the first type are described in [2].

The numerical evaluation of a series of topographs, recorded under different angles of incidence, allows for laterally resolved x–ray diffractometric measurements. As an example of application we present the investigation of the variation of the germanium percentage in a laterally structured $Ge_{0.007}Si_{0.993}$ layer on SiO_2 on Si(111). This layer has been grown by Epitaxial Lateral Overgrowth (ELO) using area selective liquid–phase epitaxy [3]. Similar systems of pure silicon on SiO_2 on Si(111) have already been investigated extensively by double crystal x–ray topography [4].

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APPLIED ASPECTS OF X-RAY DIFFRACTION INTERFEROMETRY

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The opportunities of X-Ray three crystal interferometry at the research of single crystals defective structure are demonstrated in given work:

1. Stacking faults in Si. The images of such defects in the X-Ray diffractional moire pattern is observed in a kind of gap and displacement of the moire fringe on a part of period. The value of displacement is defined by Burgers vector and system of reflecting planes. The values of stacking fault energy evaluated from moire patterns are in the range of 0,07-0,14 J / m^2.

2. Swirl - defects in Si. Discussion of probable models of lattice period variations near growth fringes is carried out on the basis of the obtained moire patterns of these defects. By selection of various models of growth fringes the satisfactory matching of growth fringes calculated images with experimental ones is obtained, as well as deformations and strains near growth fringes are quantitativly evaluated.

3. Acoustic deformations. By excitating of the ultrasonic transverse standing wave in the analyzer of three crystal interferometer the heaviest variations of moire fringes periods are found at frequencies of ultrasound close to the X-Ray acoustic resonance condition. Thus not only the degree of acoustic wave field uniformity is determined, but also local value of its amplitude in each point of crystal is evaluated.

Following structural distortions of crystals are separately considered and investigated:

- Ansambles of growth dislocations;
- Space distributions of deformations at homogeneous thermoelastic distortion of Bragg planes;
- The fields of displacement and deformations in Si at action on a crystal of external concentrated loads (space distributions of deformations in the vicinity of point of force application on the plane and wedge-shaped crystals are plotted);
- The roughness of interfaces and relaxation of straines in epitaxial systems Si-Si, Ge-Si, GaP-Si;
- Distinctions in distribution of displacement fields and deformations, arising at laser irradiation of Si surface ;
- Moire - *Pendellösung* interaction in a perfect interferometer, which arises due to interaction of two essentially different physical effects - moire and *Pendellösung*.

PATHERSON METHOD USING IN ELECTRON AND X-RAY DIFFRACTION IN REAL CRYSTAL

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Analysis of the crystal lattice image in a number of structural tasks in material science meets the obstacles connecting to limited resolution of the atom resolving electron microscopes. Due to technical and physical reasons including spherical aberration the best known microscopes have the resolution power of 1-2 Å.
This situation can be improved by using Patherson method well known in X-ray structural analysis.
Application of this method is most important for deceasion fundamental problems of microelectronics. Existence of microdefects in VLSI on the base of silicon single crystals determine the quality of the electronic devices.
As-grown microdefects in silicon single crystals can be observed by TEM and X-ray topography. We have attempted to compare Patherson maps constructing on the base of electron and X-ray diffraction patterns for silicon crystals with and without microdefects. CZ and FZ-grown crystals were used.
On the electron diffraction patterns recieving by transmission of electrons along <110> some reflections as well for matrix and also for microdefects can be seen. Patherson map for microdefect was the same that atomic models of thermodonors analyticaly constructing by two groups of researchers. This map was more bad than the atomic models because electrongrams contained a few of reflections. Under study of silicon crystals it is necessary to use 004 reflection because in another case the image will be the same than one of f.c.c. (not diamond) lattice.
Patherson map which was constructed for (110) plane of silicon after the data of X-ray diffractometer in the laboratory headed by Prof. V. I. Simonov (Institute of Crystallography, Moscow) has shown fine images of atomic pairs which can be resolved only in the best atom resolving microscopes.
Application of Patherson method is possible for observation not only of atomic columns in real crystals along the transmission direction, but also of individual lattice defects, for instance, for interstitial dumb-bells.

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X-RAY DIAGNOSTICS OF STRUCTURE PERFECTION OF SLIGHTLY DISTORTED SINGLE CRYSTALS IN BRAGG GEOMETRY

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New X-ray diffraction method for structure perfection diagnostics of slightly distorted single crystals is considered. The approach is based on measuring in single crystal Bragg geometry of the total integrated intensity of reflected beam I_R [1] and on recording of the profile of diffracted intensity spatial distribution I(x) [2]. It permits to calculate the values of coherent and diffuse components of the crystal integrated reflectivity as well as the static Debye-Waller factor exp(-L) and coefficient of additional X-ray energy losses due to diffuse scattering on defects without using a perfect reference sample.

Examples of investigation of silicon crystals containing various types of structure distortions are given. Experimental data were obtained with AgK_{a1}-radiation for different kind of symmetric reflections.

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TRIPLE-CRYSTAL DIFFRACTOMETRY IN GRAZING-INCIDENCE X-RAY DIFFRACTION

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Now, it is commonly recognized that grazing-incidence x-ray diffraction (GID) is superior to conventional x-ray diffraction techniques in studies of crystal structure of thin surface layers. The recent advances in the brightness of x-ray synchrotron sources promise a new qualitative level of GID experiments.

Up to now, the experimental techniques of GID have been confined by two conventional double-crystal schemes, where (i) the incident beam is collimated in the Bragg plane and the sample is rocked near the Bragg position [1], or (ii) the incident beam is uncollimated in the Bragg plane and the diffracted waves are analyzed depending on their exit angle to the surface [2,3]. In these schemes, the diffracted intensity is registered integrally with the diffuse scattering caused by structural defects and interface roughness. In particular, the scattering on roughness is especially strong in GID and in the case of an uncollimated incident beam [4]. Thus, an accurate interpretation of the measurements is impeded.

By analogy with conventional x-ray diffraction, the angular spectrum of x-rays reflected by the sample in GID can be analyzed with the triple crystal diffractometry (TCD), and, thus, the coherent diffraction and the diffuse scattering can be separated. We have studied the TCD in GID methodologically. The experiments have been carried out at the beamline D4 of HASYLAB/DESY. The samples were a perfect Ge crystal and a GaAs/AlAs superlattice consisting of 20 periods of 73Å GaAs and 154Å AlAs. The measurements were compared to the dynamical theory of GID in multilayers [3] and a good agreement between the theory and the experiment has been found.

Specific of GID is a complicated angular spread of the scattered x-rays in the Bragg plane and with respect to their exit angles. The analysis in either of the two planes has been implemented by analogy with the TCD in the inclined Bragg geometry [5,6] and it has been proved that the simultaneous application of the analyzer crystal in the Bragg plane and the position sensitive detector in the other plane is a minimum setting that can provide the complete solution to the problem. As a result of the study, also the requirements to the parameters of monochromator and analyzer have been determined.

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X-RAY MULTIPLE BEAM AND STANDARD HIGH RESOLUTION DIFFRACTOMETRY IN THE STUDY OF III-V EPILAYERS

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X-ray multiple diffraction, in contrast to the two-beam diffraction, occurs when several sets of atomic planes in a crystal are simultaneously or successively brought into a position to diffract an incident X-ray beam (simultaneous and successive multiple diffraction, respectively). Further on, the X-ray multiple diffraction can be coplanar (lattice vector coplanar, or wave vector coplanar) and non-coplanar [1]. Simultaneous multiple diffraction can in principle give three-dimentional information about the structure of crystals, i.e. lattice constants, lattice mismatches in layered structures and reflection phases [1-3].

Coplanar cases are interesting because of their compatibility with standard high resolution diffractometers. Based on the experimental observation of simultaneous CoKa1 (004) and (115) diffractions in some InGaP/GaAs speciments [4] a multiple beam coplanar diffractometer with a monolithic fourfold beam conditioner has been devised and constructed. Its advantage is that it gives two substrate and two epilayer peaks in (004) and (115) diffractions in one ω scan. Detailed analysis based on the relative angular positions of these peaks can give substrate lattice parameter, perpendicular and parallel mismatches, and crystal lattice misorientetions. A comparison with standard high resolution diffractometry is given as for the number of necessary measurements to obtain the required information and as for the importance of measuring the substrate lattice parameter.

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NEW POSSIBILITIES OF THE X-RAY STANDING WAVE METHOD UNDER MULTIPLE DIFFRACTION CONDITIONS

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When the Bragg conditions are simultaneously held for several families of atomic planes in a crystal, the multiple X-ray diffraction occurs . In this situation, two or more diffracted beam leave the crystal (in contrast to conventional Bragg or Laue diffraction, in which there exists a single diffracted beam). This effect only is possible at a specific crystal position with respect to the incident beam.

Until recently, the interest to multiple diffraction has been largely related to the possibility of the experimental determination of the phase of the structure factor, which essentially provides the solution to the central problem in structure analysis. The new interest to this well known effect, discovered by Ewald, is primarily caused by the advances of the dynamical theory of multiple diffraction, which predicted a number of new interesting multibeam effects, and by the instrumentational development, which made it possible to investigate the multiple diffraction of the plane waves and use its unique application capabilities [1].

In order to study the plane-wave multiple diffraction, it is necessary to collimate the incident beam in several directions and to rotate the crystal around two independent axes with high precision. [2]

New possibilities are opened by combining the multiple diffraction with the X-ray standing wave (XSW) method. The XSW method [3], based on recording of different kinds of secondary radiation (fluorescence quanta, photoelectrons, etc.) under dynamical X-ray diffraction, allows one to precisely determine the displacements of the matrix atomic planes from their regular positions, the positions of the impurity atoms, and the parameters of the artificial surface layers.

Under multiple diffraction, the beam is diffracted by nonparallel (in the general case) planes, and the XSW method allows one to obtain the information of the bulk structure of the crystal. On the other hand, the secondary radiation yield is proportional to the field intensity at the atomic planes, and the XSW permits studying the spatially modulated wave filed under multiple diffraction [4].

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STUDIES OF PROCESSES OF DIFFRACTION OF HIGH-MONOCHROMATIC GAMMA- RADIATION IN REAL SINGLE CRYSTALS.

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Diffraction of high-monochromatic ($\Delta\lambda/\lambda \leq 10^{-6}$), short-wavelength ($\lambda$=0.003nm) gamma-radiation was used to experimental study of diffraction process in real single crystals.
To check-up the modern theoretical elaborations in the field of creation of the general theory of diffraction [1-5], in additional to recent gamma-ray [6] and X-ray [7] experimental tests of Kato's statistical dynamical theory by measuring the period of the intensity beats and values of integral intensities as a function of the sample thickness or wavelength that can be applied only to highly perfect crystals, we carried out experiments on both quite perfect crystals and crystals with relatively high distortions of the crystal lattice formed during crystal growth and/or different technological operations. It was used dislocationless Si and Ge single crystals, Si undergone special purposeful influences to their crystal structure and Ge after plastic deformation. Also we investigated single crystals of quartz, which as Si has rather high degree of structure perfection, but principally different, from point of influence to diffraction process, low-dislocation defect structure.
Mainly it was used the possibility of measuring the absolute values of integral reflecting power in the condition of a high angular resolution for several orders of reflection (hkl).
So we had possibilities to realise the conditions for changing the values of static Debye-Waller factor E in all range from 1 to 0 and measuring the dependencies of E upon reflection vector for various types of lattice defects.
A detailed discussion of the applicability of Kato's statistical dynamical theory (with account of last theoretical elaborations) and other modern theories to short-wavelength gamma-ray diffraction experimental data are given.
This work is supported by Russian Foundation of Basic Research, grant 94-02-03771.

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Phase-contrast hard X-ray microtomography by Bragg-Fresnel Optics

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Recently it was found that transparent in hard X-ray (10-20 keV) range native and synthetic microobjects (10-20 microns size) mounted in a X-ray beam give an image contrast in spite of very small refraction and negligible absorption. Refraction contrast caused by Fresnel diffraction of the transmitted wave becomes possible while we are using high intensity, well collimated, small divergence and high level monochromatization beams. The information obtained from registered diffraction patterns is deficient in the correct solution of the problem of phase-contrast computed microtomography (determination of the shape, density variation, inhomogeneity, defects in microobjects). But it is possible to obtain additional quantitative information using so-called double-crystal diffraction technique where the X-ray wave formed by the Bragg-Fresnel lens and transmitted through the transparent microobject is the incident wave for the crystal-analyzer. Thus the problem of phase-contrast computed microtomography falls into four problems:

1) deconvolution of the rocking curve of the crystal-analyzer in order to obtain modulus of the Fourier spectrum of the transmitted wave;

2) phase retrieval from the known absolute value of the amplitude of the transmitted wave (image pattern) and modulus of its Fourier spectrum;

3) deconvolution of the integral equation which describes Fresnel diffraction of the wave modulated by the thickness function of the microobject;

4) standard back-projection algorithm as in plain tomography to find unknown two-dimensional shape and volume variation of refractive index in the microobject.

ANOMALOUS NORMAL SURFACE STRESS IN THIN FILMS OBSERVED WITH HIGH-RESOLUTION SYNCHROTRON DIFFRACTION

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A novel technique which measures the normal stress for the top 20 atomic layers of a thin film will be presented. This technique determines the variations in strain anisotropy as a function of depth in thin films using high-resolution synchrotron diffraction. The residual normal (σ_z) and azimuthal stresses (σ_x and σ_y) in the top 5 nm, top 10 nm, and through the entire film thickness in several Mo films was investigated. The Mo films had nominal thicknesses of 50 nm, 100 nm, 170 nm, and 270 nm, and were deposited by direct current magnetron sputtering onto Si (100) substrates. The magnitude of the normal stresses were $-1.00 \pm .45$ GPa and $0.02 \pm .45$ GPa in the top 5 nm for the 50 and 100 nm thick films, respectively. However, the normal stresses in the top 5 nm of the 170 and 270 nm films were much larger, and on the order of -3 GPa. The normal stresses were nominally zero for all of the films when the scattering volume included the entire film thickness, as expected. X-ray pole figures, conducted on a 12 kW rotating anode source, showed the 50 and 100 nm films to be random and polycrystalline, and the 170 and 270 nm films to possess a <110> out-of-plane texture. The depth sensitivity for the strain measurements was obtained by varying the incidence angle of the incoming monochromatic synchrotron radiation near the critical angle for total external reflection for Mo. At each penetration depth, at least six diffraction peaks were collected and the entire strain tensor was determined by a least squares methodology. The origins of the normal stress will be discussed. The role of surface roughness and the possibility of modulus variations near the surface will also be considered.

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GRAZING INCIDENCE DIFFRACTION FROM $LINBO_3$ UNDER EXCITATION OF SURFACE ACOUSTIC WAVES

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Due to its optical and piezoelectric properties $LiNbO_3$ represents a material with promissing applications in the field of optoelectronic devices. Under excitation of an ultrasonic surface acoustic wave (SAW) it can be turned into a wave guide or light deflector. Y- cut $LiNbO_3$ single crystals have been equipped with interdigital electrodes to implant a SAW along the hexagonal c direction running parallel to the surface. It is the aim of this work to study the diffraction behaviour of a vibrating crystal and to characterize the vibrations as a function of depth and of the applied excitation voltage.

In recent years we have developed the method of depth resolved grazing incidence diffraction (GID). It was used to measure the (006) reflections under the condition of grazing incidence exploiting synchrotron radiation. Depth sensitivity is obtained by recording the diffracted intensity along grazing exit angles by use of a position sensitive detector. Due to the scattering geometry the influence of longitudinal, SAW induced displacements on the scattering process is probed as a function of depth.

We find that the exit angle resolved intensity profil is dominated by a surface Bragg peak with dynamical broadening which becomes sharper and higher with the applied voltage. Simultaneously the integrated intensity increases. The results have been fitted by a heuristic approach for the scattering theory since for the GID case, a dynamical theory including increasing distortions is not available in the literature. We demonstrate that the shape and the measured scaling factor of the exit angle resolved intensity as a function of the angle of incidence and the applied voltage can perfectly be fitted by solely introducing a static Debye Waller Factor which reduces the structure factor. The scattering process transforms from dynamic to kinematic. The measured avarage static displacement increases linearly with the excitation voltage and the intensity scales with the extinction length like τ^2. In the near surface region probed in the experiment ($\leq$ 2000Å) the amplitude of the SAW is not attenuated.

New Methods for Depth Profiling of Heterostructures by X-ray Diffraction

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High resolution x-ray diffraction is commonly used for the characterization of semiconductor heterostructures. The typical penetration depth of the x-rays is about a few microns and normally covers the whole layer stack. For extremely asymmetric reflections it can be significantly reduced so as to perform diffraction near the surface and by this a characterization of only the top layer(s). A continuous variation of the penetration depth makes it possible to perform depth profiling.

The availability of a powerful depth profiling will be of great importance for the characterization of layer structures. For example, a peak splitting or broadening is often observed in rocking curves of ternary or quaternary layers. This is probably caused by a variation of the composition in dependence of the depth due to small unintended drifts of the growth parameters during epitaxial growth. To avoid such problems one has to determine the layer composition in dependence of the layer depth. In order to analyze the structural parameters of layers in dependence of the depth we investigated II-VI-layer structures grown by molecular beam epitaxy (MBE).

In principle a continuous variation of the penetration depth can be achieved by a variation of the absorption length. In case of extremely asymmetric reflections, the problem is to keep the absorption length constant while recording diffraction profiles like rocking curves or q-scans. We will present two new approaches to solve this problem. The first approach is based on synchrotron radiation with the possibility to change the wavelength, the second is based on a tricky variation of the diffraction geometry, which can be combined with conventional laboratory x-ray sources. By these methods, rocking curves and reciprocal space maps can be measured with constant and small penetration depths of the radiation. The first results of these two new methods applied to II-VI-layer structures will be presented here.

ENERGY DEPENDENCES OF INTEGRATED REFLECTIVITIES IN THE LAUE CASE OF X-RAY DIFFRACTION BY IMPERFECT CRYSTALS

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The aim of present work is to investigate the sensitivity of both total integrated reflection power (TIRP) of thin single crystals containing homogeneously distributed defects and partially separated coherent and diffuse components of TIRP with respect to defect characteristics.

The measurements were performed by using the triple crystal X-ray diffractometer in both double- and triple-crystal mode of the diffraction scheme with several characteristic wavelengths. The use of the triple crystal mode in measurements of integrated reflectivities of imperfect crystals gaves the possibility to suppress partially in a controlled way the coherent or diffuse component of TIRP (in dependence on the choosen variant of measurement scheme). Thermally treated samples of Czochralski-grown Si single crystal were investigated.

Theoretical analysis and treatment of the measured energy dependences of TIRP and its components in the Laue case of X-ray diffraction by thin crystals with microdefects have been carried out by using the analytical expressions obtained by the authors in present work as the generalization of the previous formulae [1]. The different behaviour of coherent and diffuse components of TIRP versus radiation wavelength as well as the strong energy dependence of the coefficient of extinction due to diffuse scattering, which is involved in both coherent and diffuse extinction factors in the corresponding components of TIRP, cause the high sensitivity of the energy dependences of TIRP and/or its components (in dependence on their ratio) to defect characteristics.

The obtained results show that the energy dependences of TIRP and its components can be used to rise the reliability and precision of the nondestructive quantitative diagnostics of structure imperfections in real single crystals.

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On the Interpretation of X -Ray Standing Waves Data in Vibrating Crystals

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It is well known that X-ray Standing Wave (XSW) method is based on the measurements of the fine structure of the yield of the secondary process in the angular vicinity of the X-ray reflex,[1]. Under the condition of dynamical diffraction there is an unique possibility to investigate the position of the resonant atom inside the crystal, because the yield of the secondary process is directly proportional to the intensity of the X-ray wave field which has the period equal to the interplanar distance. It means that X-ray wave field provides us with a perfect scale with the period about 1-10A.

Here we consider the new possibility of XSW method in Bragg case in vibrating crystal, i.e. crystals disturbed by longitudinal ultrasonic vibrations with wave vector $\mathbf{K}_s$ and the amplitude of displacement, hw directed along to the normal to the crystal surface. Due to the one dimensional distortion one can present a vibrating crystal as a periodic set of slides with constant value of interplanar distance. Each of the periodic set gives rise to additional reflex (usually called satellite) corresponding to the diffraction with wave vector $\mathbf{h} + n\mathbf{K}_s$, where h is the initial diffraction vector, $\mathbf{K}_s$ wave vector of the ultrasound. The main reflex with diffraction vector h is formed by the undisturbed part of the crystal. With the increasing of the amplitude of ultrasonic wave the fraction of the undisturbed part decreases and gives a share to the part corresponding to the first-order satellites. The complex process of the splitting of the crystal into the set of the slides is described by the well known formulae defining the extinction length, Λ_m, and structure factor, $\chi_{h,m}$ of the main reflex and satellite of the m-order, [2],

$$\chi_h^{dis} = \sum_{m=-\infty}^{\infty} \chi_{h,m} \exp(\mathrm{im}Kx), \Lambda_m = \Lambda/|J_m(\mathrm{hw})|, \chi_{h,m} = \chi_h i^m J_m(\mathrm{hw}),$$

where $J_n(\mathrm{hw})$ are the n-th order Bessel functions of real argument and, Λ is the initial extinction length.

For the sake of simplicity we consider only the case of so-called "high frequency" ultrasonic wave, when the angular distance between the satellites is a rather large to distinct them. Under these conditions the vibrating crystal provides us with a unique technique when instead of the one rocking curve you can measure a set of rocking curves corresponding to different satellites and moreover to vary the extinction length each of them within the limits from Λ to infinity.

It means that instead of the scale with fixed value of period you gain a set of scales with varying period and phases.

Specific features of the XSW method in vibrating crystal, experimental data and computation procedure are discussed.

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CHARACTERIZATION OF EPITAXIC HETEROSTRUCTURES, SUPERLATTICES AND GRADED LAYERS USING THE STATISTICAL THEORY OF X-RAY DYNAMICAL DIFFRACTION

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The high resolution X-ray diffraction techniques were used for the study of heteroepitaxial layers, superlattices and graded crystalline systems. The experimental investigations were performed on the double- and the triple diffractometer. The coherent component was separated using a procedure based on θ-2θ scanning of the sample crystal and analyzer crystal. In the double- and the triple diffraction schemes the primary beams of CuK_{α_1} radiation were collimated and monochromatized by the asymmetric reflection from a perfect Ge crystal. The heteroepitaxial laser structure and *AlGaAs* graded layer were grown on a thick *GaAs*(001) substrate by metal organic chemical vapor deposition.

The statistical theory of x-ray dynamical diffraction was applied to analyze rocking curves. The coherent and incoherent parts of scattered intensity were taken into account. We have considered the laminae model for calculation of x-ray diffraction curves from a multilayer structure and graded layer.

The structural and compositional quality of heteroepitaxial structure and graded layer were determined from profiles of rocking curves. Using calculated procedures by means [1] we have found the depth-dependent compositional nonuniformity, the depth distribution of the static Debye-Waller factor and average size of defects.

We have developed the theory of X-ray diffraction from superlattices with the fluctuation of a period and containing microdefects in sublayers. The experimental results for *GaAs / InAs* -superlattices were analyzed on the basis of numerical calculations.

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X-RAY DIFFRACTION INVESTIGATIONS OF STRUCTURAL CHANGES IN InSb AND Si CRYSTALS, IRRADIATED BY HIGH-ENERGY ELECTRONS

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At irradiation of elementary semiconductors, such as Si and Ge, by electrons, two types of point-like defects, vacancies and interstitial atoms, are possible. Number of possible kinds of defects in binary compounds is much greater. The purpose of given work is the crystal lattice distortions research, which results from a high-energy irradiation of single crystals by electrons: for Si - energy of electrons E=10 MeV; for InSb - E=18 MeV. Dozes are from 10^{14} up to 10^{17} e/cm/s).

The analysis of space distributions of intensity over the Borrmann fan I (x), as well as research of changes of dependence character of absolute values of integral reflecting ability for given crystals from order of symmetric reflexes (333), (444), (555), (777), (888) in $MoK_{\alpha 1}$-radiation has been performed. High-energy irradiation by electrons is found to improve, as a rule, a degree of structural perfection of crystals. Such conclusion is confirmed also by the extinction length variation, which is determined from X-Ray acoustic researches. We shall note, that the difference of reflecting ability of the opposite surfaces of the crystal increases with the irradiation doze increase. The exit surface of the sample has appeared more perfect than directly irradiated one. This phenomenon is explained by the presence of strongly distorted areas in the subsurface layers along the direction of trajectories, on which expeled atoms with large energies move. For refinement of the opportunity of such structural changes both in volume of the crystal, and in subsurface layers also a two crystal spectrometer with installation of a researched crystal in a scew asymmetric geometry of diffraction is used. The azimuthal scanning of the crystal around of a diffraction vector has allowed to investigate and to confirm structural changes both in volume and in thin subsurface layers of crystals.

SYNCHROTRON RADIATION X-RAY MULTIPLE DIFFRACTION IN THE CHARACTERISATION OF THE AlGaInAs/InP(001) STRUCTURE.

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The successful application of the X-ray Multiple Diffraction technique using synchrotron radiation to characterise an AlGaInAs layer grown on InP(001) is described.

The multiple diffraction (MD) phenomenon is systematically obtained in the Renninger geometry using a collimated beam which provides the so called Renninger scan (RS). In this scan, a single crystal is rotated about the normal to a set of diffracting planes [primary (h_p, k_p, ℓ_p)] while the diffraction from these planes is monitored. Under rotation, other planes [secondary (h_s, k_s, ℓ_s)] diffract simultaneously with the primary. The transfer of energy between the primary and the secondary beams occurs within the crystal through the MD coupling reflections (h_p-h_s, k_p-k_s, ℓ_p-ℓ_s). Surface secondary reflections are very special MD cases since they allow the primary beam to be diffracted almost parallel to the sample surface in a extremely asymmetric condition. The occurrence of the layer and substrate lattice interactions in the MD for heteroepitaxial structures is fundamental for characterising these structures since information is provided through modulation of the Renninger Scan Intensities (MORSI) (Greenberg and Ladell, Appl. Phys. Lett. (1987), 50, 436). Extra dips appear in the substrate RS due to the diffraction of the incoming and primary beam intensities (when both cross the layer) by epilayer planes in certain orientations.

High resolution RS were obtained of InP(006) bulk and substrate and, of an AlGaInAs layer (2 μm) on high resolution set-up developed at station 7.6 of the Daresbury Laboratory. The beam incident on the sample is extremely monochromatic and collimated (2 arcsec in both horizontal and vertical divergence). The layer was grown by Metal Organic Vapour Phase Epitaxy (MOVPE) and its stoichiometry was determined by gap-energy and lattice mismatch as $Al_{0.304}Ga_{0.172}In_{0.524}As$.

The RS can provide: I) the used wavelength from the surface secondary peak position; ii) the layer parallel lattice parameter from the tetragonal distortion measured in the 6-beam case at χ=90° and from the MORSI features measured in the substrate RS; iii) the relative tilt between the substrate and layer lattices from the mirror symmetry positions in both layer and substrate RS; iv) the mosaic spread in plane of the layer and the substrate from the surface secondary peak profile. Furthermore, the layer RS simulated with the MULTX program shows a very good agreement (reliability of the order of 0.085) with the corresponding experimental data.

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THE DEVELOPMENT OF X-RAY STANDING WAVE METHOD FOR NANOSTRUCTURE CHARACTERIZATION

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X-ray standing wave (XRSW) technique is based on the excitation of secondary radiation at the interaction with the crystalline structure of the periodic X-ray wave field formed under the dynamical diffraction conditions [1]. This surface sensitive high resolution X-ray method has been developed for structure characterization of near perfect mainly semiconductor crystal surfaces, interfaces, epitaxial films and heterostructures.

For investigation of long range order systems (organic monolayers, electrochemical interfaces etc.) artificial long period XRSW generators - layered synthetic microstructures, Langmuir-Blodgett films and mirror surfaces under total reflection condition have been applied [2].

The achievements of nanotechnology allow to manipulate with ultra-thin layers constructing on their base absolutely new nanometer-scale materials and devices. The present paper demonstrates how XRSW method can been adjusted for characterization of super thin solid overlayers (crystalline and disordered, organic and inorganic) [3,4] and structures on their base (consisting of several layers and periodic) [5]. It is accompanied by the analysis of dynamical diffraction phenomena in various multilayers and interference effects developing in layered systems and leading to XRSW formation inside and above X-ray wave-guide structures [6]. The main attention is paid to the situations when structure and geometrical parameters as well as optical characteristics of ultra thin layers may be obtained.

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DYNAMICAL THEORY OF COHERENT EXCITATION OF X-RAY CASCADE BY SYNCHROTRON RADIATION

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Coherent excitation of cascade of X-Ray transitions (XC) [1] by synchrotron radiation (SR) in perfect crystals is theoretically investigated in the framework of dynamical diffraction theory [2]. The angular distribution of the XC last transition radiation is described by equation

$$\mathrm{rotrot}\mathbf{E}(\omega)+(\omega/c)^2\varepsilon(\mathbf{r})\mathbf{E}(\omega)=\chi(\mathbf{r})\mathbf{E}_s \qquad (1)$$

where $\mathbf{E}(\omega)$ and ω are the electric field and frequency of the wave emitted in the XC last transition $\varepsilon(\mathbf{r})$ is the dielectric tensor of the crystal at the frequency ω, $\mathbf{E}_s$ is the SR electric field at the frequency of the XC highest level and $\chi(\mathbf{r})$ is the "inelastic susceptibility" of the crystal determined by the characteristics of the XC, structure and dynamical properties of the crystal.

In the thin crystal limit eq.(1) describes the kinematical results [1] and, in particular, shows that only the angular distribution of the last quantum of the XC is strongly influenced by the symmetry and size of the crystal unit cell. For instance, diffraction maxima are possible in the angular distribution of the last transition quanta.

In thick crystals dynamical effects are present. There are a finite angular width of diffraction maxima, pendelum beats versus the sample thickness, complicated polarization properties of the last transition quanta etc. The fine structure of the angular distribution of the last transition quanta are calculated. Special attention is paid to the role of the phase-matching conditions in the angular distribution of the last transition quanta close to forward direction. The optimal conditions for experimental observation of the examined effects are discussed. As a specific example the case of two X-ray transitions cascade in tungsten is analized.

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X-RAY STUDY OF GaAs/Ge HETEROSTRUCTURES : RELATIONSHIP BETWEEN INTERFACIAL DEFECTS AND GROWTH PROCESS.

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The GaAs/Ge system is characterized by a very low misfit (7.6 10^{-4} at room temperature), so dislocation densities are very low. In this way, X-Ray topography has several advantages to study the early stages of strain relaxation process. At first, it is a large scale, non destructive technique, allowing to observe an entire wafer. Then, two complementary kinds of images can be obtained : X-Ray transmission (Lang setting) gives topograms corresponding to the contribution of both the film and the substrate; X-Ray reflection (Berg Barrett setting) allows to separate diffracted peaks corresponding to (hkl) planes from the layer and the substrate, and two separated images can be obtained. The comparison between both diffracted modes allows to evaluate the dislocation density in the layer and in the substrate. Finally, the Berg Barrett setting allows us to measure the tetragonal parameter of the film, so we can get information about strain and relaxation in the layer.

Two kinds of samples have been studied :

- GaAs films grown by CSVT ("Close-Spaced Vapor Transport") on (100) Ge substrates at T_g = 750 °C.
- GaAs films grown by MOVPE on (100) Ge substrates, 6° off towards [110], at T_g = 600 °C.

All the measurements indicate that the GaAs layers are highly metastable. Transmission and reflection topograms show that the experiment critical thicknesses for first misfit dislocations development depend on growth process, and are several times higher than the theoritical one. Moreover, the density of the dislocations networks observed at further stages is not high enough to give a complete relaxation of the layer. These misfit dislocations are much longer for the samples grown by CSVT than for the samples grown by MOVPE, whereas they are nucleated for smaller thicknesses in the later.

These results are strongly dependent on growth conditions : growth process and temperature, cooling and growth rates, structural quality of the Ge substrate. Indeed, nucleation and propagation of dislocations in semiconductors depend on temperature and stress, so a lot of parameters must be taken into account in comparative studies. Of course, such studies are well completed by TEM observations.

The detected defects have been correlated with the morphology of the thinnest layers grown under the same conditions for very short times. The surface analysis has been assessed by using atomic force microscopy (AFM).

EFFECT OF MULTIPLE DIFFUSE SCATTERING ON LAUE X-RAY DIFFRACTION BY CRYSTALS WITH MICRODEFECTS

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The general theory of X-ray diffraction in statistically distorted crystals should take into account: (1) the renormalization of the coefficients of the dynamical theory due to crystal lattice distortion (static Debye-Waller factor, diffuse absorption), (2) multiple diffuse (incoherent) scattering, i.e., the phenomenon of secondary extinction, and (3) the dynamical (coherent) diffraction of diffuse-scattered waves [1].

In the case of double-crystal recording of the angular distribution of scattered intensities the neglect of the dynamical diffraction of diffuse-scattered radiation is justified by the fact that for defects of radii r much lower than extinction length Λ the angular width of the diffraction range is much lower than the width of the angular distribution of diffuse scattering [2-4]. At the same time, allowance for secondary extinction, seems to be mush more important [4], because the angular widths of diffuse scattering, secondary extinction, and diffuse absorption are approximately the same.

The effect of multiple diffuse scattering on the angular distributions of reflection, transmission, and integrated reflection is considered. The space-angular dependence of the dynamical scattering are defined by two systems of statistically averages equations for the coherent components of the wave amplitudes, and for the intensities of diffuse-scattered waves [3, 4]. The uniform distribution of microdefects in the crystal volume is assumed. The general results are considered on a model of defects with the pair correlation function of Kato in the exponential form.

On the basis of the calculations performed, the behavior of the reflection and transmission curves is analyzed as a function of the static Debye-Waller factor in the range from $f = 1$ to $f = 0$, defects radii $r \leq 0.1\Lambda$, and crystal thickness $\mu t < 6$. It is shown, that the influence of the multiple diffuse scattering on Laue X-ray diffraction is essential even at sufficiently perfect single crystals with $f \sim 0.95$. This effect results in a decrease of intensity in the reflection direction and on increase and considerable broadening of transmitted intensity in comparison with those calculated in the kinematical (with respect to diffuse scattering) approximation.

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POSSIBILITY OF SOLVING THE INVERSE PROBLEM OF X-RAY DIFFRACTION IN LAYERED STRUCTURES IN THE DYNAMIC CASE

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For analysis of layers of any arbitrary thickness (in the case of a crystal structure with disturbed-layer thickness $T>\Lambda_{ext}$, where Λ_{ext} is the X-ray extinction length) the Born approximation is inapplicable, and it is necessary to take account of the dynamic effects of scattering. In this case restoration of the complex reflecting capability (CRC) profile (CRC depends on the structure factor $\sigma(z)$ and the elastic displacements u(z) of the lattice sites) from the known amplitude reflection coefficient is a more complex problem /1,2/. This work deals with this problem, namely, to solve the inverse problem of diffraction scattering of X-rays on the basis of the dynamic theory of Bragg reflection from a layered crystal structure.

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Improved dynamical theory for x-ray reflectivity of ideal crystals of finite size at low and high incidence angles

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We present the exact analytical equations of the dynamical theory within the Laue-Ewald formalism for the two-beam case valid for the x-ray Bragg diffraction on ideal crystals of finite thickness. In our calculations no approximations are made, which are usually used in the "conventionally" dynamical theory in order to solve the secular equation. In particular, in our calculation are taken into account: i) the asymptotic sphericity of the dispersion surface and all the four solutions of the secular equation; ii) the difference between electric and displacement fields; iii) the boundary conditions of continuity of the tangential components of the electric and magnetic fields at the two crystal-air interfaces. With the new equations derived it is possible to describe the interaction of the x-ray beam with the crystal in a dynamical way in the whole angular range from 0 to 90°. In fact, the theory describes dynamically the Bragg diffraction as well as specular reflectivity near and below the critical angle in terms of four primary waves and four waves diffracted in the crystalline medium. It should be also noted that our formalism is valid for symmetric and asymmetric reflections both near the Bragg condition and at the far tails of the Bragg peaks. Moreover, the amplitude of the transmitted wave from the lower crystal surface, both in conditions of specular reflectivity and Bragg diffraction, is provided by the same equation. Our equations can be also applied for describing Bragg diffraction of x-rays at small glancing angles of the order of the critical angle, i.e. in conditions where specular reflectivity contributions can be not neglected.

INDEX OF REFRACTION AS A DYNAMICAL VARIABLE IN THE SELF-CONSISTENT EM SCATTERING. By D.S. de Vasconcelos and W.A.Keller, Instituto de Física, Universidade Federal da Bahia, Salvador, Ba., Brazil.

The self-consistent treatment, developed initially for X-ray diffraction in perfect crystals, is used to define the index of refraction as a dynamic parameter even in a region where the Bragg law is not valid. This index of refraction is a straight-forward consequence of using the self-consistent mode of interaction developed between light, represented by EM field, and matter, by a set of vibrating dipoles. In this mode of scattering the EM field forces dipole oscillations, whose field must accounted for as well as external field, composed of two waves, the incident and the reflected. The dipole, contribution allows the energy of the system (considered without absorption) to be conserved.

Refraction and absorption are treated separately and appear as 'macroscopic' type phenomena in the directions parallel to the scattering planes of dipoles and 'microscopic' phenomena in the direction normal to them. The last property is a direct result of the approach developed herein which treats the plane of dipoles as an EM plane wave transformer, altering in a steplike manner the amplitude and the phase of both interacting waves. The change in phase mentioned causes the direction of energy flow to be different from the direction if the actual wave vector which gives rise to the appropriate index of refraction, which in turn is different of either of the interacting waves. The second reflected wave, though it appears as a macroscopic entity only in some special cases, always has an important role in microscopic EM fields (evaluated at the dipole level).

The calculated values of index of refraction for Si single crystals within this model are smaller than unity for X-rays and larger than unity for light of longer wavelengths. This result appears for both states of polarization and does not depend on absorption. In the intermediary region, for given charge density and radiation wavelength, some critical point can always be found for which the index of refraction is exactly equal to unity.

Asymmetry effects in the plane-wave approach of the statistical dynamical theory

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The statistical dynamical theory of diffraction by crystals containing randomly distributed microdefects has been formulated by Kato (1980), using a spherical-wave approach. A basic point is that the statistically averaged wave-amplitudes, which represent the coherent diffracted waves in the Bragg and in the forward directions, suffer an attenuation (additional to usual absorption) due to gradual transformation into incoherent (diffuse) intensities. It has been later shown by Bushuev (1989), using a plane-wave approach, that this attenuation depends on the angular departure from the exact Bragg position of the crystal. This plane-wave approach was based on a one-dimensional form of the Takagi-Taupin equations, in which the deformation field of the crystal is taken as a function of position along the normal to the crystal surface. In the present treatment, this one-dimensional assumption is not used; the deformation field is taken as a function of 2 coordinates (along the incident direction and along the direction of diffraction). In the case of nonsymmetrical Bragg or Laue geometry, the attenuation terms in the propagation equations of the coherent waves are then found to be different from those of the previous formulation of Bushuev; the consequences of these differences, which disappear in the case of symmetrical geometry, will be discussed.

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MODIFIED BRAGG LAW FOR HETEROLAYER STRUCTURES.

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X-ray diffraction from stratified structures as multi-epilayers and multi-superstructures an a substrate or not is analyzed by approach developed for self-consistent EM scattering by a plane of dipoles. The introduced concept of a scattering unit for which a rigorous vectorial waves solution has been found for σ- and π-polarizations is generalized for any type of the inhomogenous structure by a demand of continuity of the EM field vectors between all the units. Diffraction profiles for selected structures which can only be handled by some hybrid kinematic-dynamic types of treatment are obtained without any added effort within the same approach.

For infinite multi-superlattice structures the diffraction profiles show features such as a sequence of tophat curves for satellite reflections with the respective total reflection depending in a critical way, on the charge densities of the component units, their structure factors and the polarization state of the incident radiation. This dependence is found of the same type as that for the main peak and is preserved even in cases when respective TRR is reduced to fractions of the arcsecond. The latter parameter, for a given order of reflection, depends on composition, homogenous layer thickness, charge density value and inter- and intra-layer distances between scattering units.

The finite multi-superlattice and multi-epilayer structures display diffraction patterns of a kinematic type. A continuous transformation of the diffraction-profiles from kinematic to dynamic type can occur as a result of a simple change of the numerical value of two parameters, which depend on structure-components, such as the layer thickness and the charge density of the respective component.

An attempt is also made to formulate a modified Bragg law version for the satellites valid for the multilayer superstructures where the appropriate refraction indices are intrinsically included into the scattering model.

INTERPRETATION OF REFLECTION CURVES FROM IMPERFECT SINGLE CRYSTALS WITH MICRODEFECTS

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The conventional use of the method of "integrated" intensities of the diffus scattering (DS) [1] is based on the kinematical approximation for the D intensity. This approximation is successful in obtaining the defect characteristic from tails of the reflection curves (RC) measured by a double-crysta diffractometer at deviation angles far enough from the total reflection range [2] However, such approach become invalid when the defect radius R_0 is comparabl with the extinction length Λ and DS scattering must be treated dynamically.

The aim of present work is to develop, on the base of the dynamical theor of X-ray scattering by real crystals [3, 4], the theoretical model describin correctly both coherent and diffuse components of RC in the whole angular rang including the total reflection range (see also [5]).

The analytical expressions for the coherent and diffuse components of R(have been firstly obtained for X-ray diffraction by single crystals containin randomly distributed microdefects. The more detailed account of dynamical effect in DS has been allowed to give the rigorous quantitative description of the diffus component of RC in total reflection range and its near neighbourhood. Th coefficient of extinction due to DS being involved in both coherent and diffus components of RC has been explicitly connected with parameters of various-typ microdefects.

To test advantages of the model, the measurements of RC's, from thermall treated Si single crystals have been carried out by using the high-resolutio double-crystal X-ray diffractometer. As follows from the analysis of the RC's, th model allows to rise the informativity of RC measurements, particularly, in th case of defects with large radii $R_0 \gtrsim \Lambda$.

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GRAZING INCIDENCE DIFFRACTION OF NARROW X-RAY BEAMS.

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In X-ray diffraction problems it is usually supposed that incident as well as diffracted beams have infinite transversal dimensions, i.e. they could be presented by means of plane waves. That approximation holds for the incident beam if wavelength $\lambda << a$, where a is the transversal dimension of the incident beam. In the X-ray band it is always true. However for diffracted beam that approximation may prove to be insufficient. If dimension of the incident beam is comparable to the extinction length $L_{ex} >> \lambda$ the effects due to transversal finiteness of the beam may result in distortion of the intensity distribution of the diffracted beam. Really finite width a of the incident beam gives rise to effective transversal component $q \sim 1/a$ of the wavevector k_0, which results to additional deviation from the Bragg angle by the value $\sim \lambda/a$. If that value is comparable to the angular width of the rocking cutrve $\sim |\chi_h|$ (χ_h - fourier component of the polarizability) then it causes considerable change of the diffraction conditions.

We investigated the effect of the finiteness of the incident beam in conditions of grazing incident diffraction by numerical and analytical methods. Special attention was paid to the intensity distribution of the diffracted wave along the surface. The knowledge of the intensity distribution is necessary for correct interpretation of grazing incidence XRSW experimental data.

X-RAY GRAZING-INCIDENCE MAGNETO-OPTICS UNDER RESONANT SPECTRAL CONDITIONS

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Giant grazing-incidence polarization phenomena at resonant reflection of soft X-rays from rare earth magnetic surface layers, films and superlattices have been examined theoretically. The unique combination of the grazing-incidence geometry and the 3d-4f atomic resonance with the well-known resonant enhancement of magneto-optical activity of rare earth elements near M absorption edges is shown to result in the strong dependence of grazing-incidence reflection amplitudes on the polarization state of an incoming X-ray beam and the magnetic state of a reflecting surface layer. Near the critical angle of total external reflection, the corresponding relative magneto- and polarization-sensitive variations of X-ray reflectivity and Kerr effect values may reach of about 80% and are easily detectable. At small glancing angles, the Kerr effect behavior is unique in relating not only to the sharp distinctions between the π and σ reflectivities, but also to the large difference in phase between the π and σ reflection amplitudes.

All this, taking into account the small tunable penetration depth (1-200 nm) of soft X-rays under discussed condition, allows us to put forward the "double-anomalous" magneto-optics as a fruitful method for determination of the depth profiles and interface distributions of magnetization in magnetic films and superlattices with a high degree of accuracy. For example, as the depth profile consists in the 90 ^{o}- rotation of a magnetization vector in a surface layer, a reflectivity change of a few percent may be observed when the width of the layer varies over 10%. The values of the basic structural magnetic and non-magnetic parameters of superlattices are easily derived from reflectivity and Kerr effect values as well. A new method to attack the problem of determination of these parameters from reflectivity measurements based on the direct use of the fundamental Bloch solutions of the Maxwell's equations is developed.

New optical elements with magnetic control for synchrotron radiation beamlines using polarization-sensitive "double-anomalous" reflection from rare earth magnetic films and superlattices are discussed. Such elements - polarization filters, convertors and switches - provide high conversion and filtration efficiency with intensity losses of about 30%.

SOME THEORETICAL ASPECTS OF X-RAY SCATTERING IN STRATIFIED MEDIA

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Layered synthetic microsrtuctures and molecular Langmuir-Blodgett films provide the unique possibility of developing x-ray-UV optical devices analogous to those used in the visible range. These structures are in fact artificial diffracting systems consisting of layers with uniform thickness and density, having parallel interfaces. Recursive formalism based on the solution of the Fresnel equations on each interface and developed by Parratt in [1] is common approach to the treatment of X-ray scattering and X-ray fluorescence yield from multilayer mirrors for all angles of Bragg and total external reflection.

This paper presents an analytical expression for the X-ray intensity reflected from a periodic stratified media that has been obtained by solution of difference Fresnel equations. The dymnamical theory of the propagation of X-ray waves through the multilayer system based on Parratt recursive formalism is considered. Its equivalence to the dymnamical theory of Darvin-Prins is discussed in details.

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Dynamical and Kinematical Theory of Grazing Incidence Diffraction

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Although it is well-known that for small incidence angles the dynamical theory approaches the kinematical one, in the case of grazing incidence diffraction we find some considerable differences between both. Both these theories are applied to experiments with samples of different crystalline perfection at the surface. For comparison with the experimental data it was neccessary to modify the conventional theories so that scans along different directions within reciprocal space are able to be simulated, as well as any deviation from the exact Bragg point (for example by large lattice mismatch).

Distinct differences can be seen in the region of the critical angle for total external reflection, α_C, (up to about 2 α_C) near the exact Bragg position. From the dynamical theory one expects an overlap of the signals coming from the anomalously strong and weak wave fields. This has remained practically unobserved even for clean semiconductor wafers. Most samples have distinctly poorer crystalline quality at the surface than in the bulk, so that kinematical calculations and fits are mostly justified. In addition the kinematical theory allows the simulation of diffuse scattering in grazing incidence diffraction.

EFFECT OF ROUGHNESS ON GRAZING-INCIDENCE X-RAY DIFFRACTION

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As has been shown by many experiments, grazing-incidence x-ray diffraction (GID) can provide extensive informations on the structure of thin surface layers of crystals. However, by analogy with x-ray specular reflection experiments, a strong effect of surface and interface roughness is expected in GID, since the x-rays graze along a crystal surface. That can give rise to a change in diffracted intensities and to a considerable role of diffuse scattering. Thus, one has to distinguish between the effects of interface roughness and crystal lattice defects on GID.

A theory of diffuse scattering on correlated and uncorrelated interface roughness in multilayers (ML) under GID will be presented. Our model is based on the matrix dynamical theory of GID in ML [1, 2] and the distorted wave Born approximation [3]. The theory is general for GID and extremely asymmetric x-ray diffraction and it is applicable in case of ML with normal lattice strains.

The contribution of the diffuse scattering to GID intensities taken in different experimental schemes will be analyzed and possible ways to separate the diffuse scattering from the coherent signal in GID experiments will be discussed.

The theory will be compared with the experimental GID data [4] taken from Ge crystals with flat (polished) and rough (etched) surfaces. A good applicability of our theory to the explanation of the observed intensities will be demonstrated.

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A DYNAMICAL DIFFRACTION APPROACH TO GRAZING-INCIDENCE X-RAY DIFFRACTION BY MULTILAYERS WITH LATERAL LATTICE MISFITS

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In recent years, interest in studies of semiconductor superlattices (SL) and multilayers (ML) has permanently grown up. The X-ray grazing-incidence diffraction (GID) method provides information on thin sub-surface layers as thin as $1-100$nm. The method is depth-selective because the penetration depth of the probing X-ray can be tailored choosing the incidence angle smaller or larger than the critical angle of total external reflection. Furthermore, the in-plane lattice parameter of crystal layers can be measured directly [1] and the degree of crystalline disorder (amorphization) and interfacial roughness can be studied [2].

A matrix approach to the description of GID in multilayers (see e.g.[3,4]) can be used as a theoretical basis of quantitative data analysis. Up to now its application required that the lateral lattice parameters of the sub-layers should coincide. In the present report we propose an extension of the matrix theory of GID which should be applicable to systems containing a lateral lattice mismatch. As a simplest case we consider a ML consisting of two types of layers with different, but constant lattice parameters. Our model can be applied to several kinds of structures: (i) samples like SIMOX where the difference in the lattice spacings is accommodated without developing misfit dislocations or 3D domains, and (ii) completely relaxed two-component MLs or SLs with relatively thick layers where the contribution of scattering at strain fields of misfit dislocations located near the interfaces can be neglected, as compared to the scattering at the other parts of the layers. Our approach is based on the dynamical diffraction theory which is strongly suggested by the permanently increasing quality of MLs. The theoretical model is illustrated by several numerical examples. The limitations and possible extensions of the theory are also discussed while these examples are analyzed. The aim of the work is to initiate new high resolution experiments using GID, like the studies of the initial state of lattice relaxation in strained superlattices, as example.

[1] Marra W C, Eisenberger P and Cho A Y 1979 *J. Appl. Phys.* **50**, 6927
[2] Stepanov S A and Köhler R 1994 *J. Appl. Phys.* **76** 7809
[3] Stepanov S A 1994 *Kristallografiya* **39**, 221
[4] Stepanov S A, Pietsch U and Baumbach G T 1995 *Z. Physik* **B 96** 341

Calibration system for x-ray microscopes

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One of the main problems in using x-ray microscopy as a tool for quantitative measurements is the problem of the determination of x-ray beam parameters. It is especially important when a relatively complicated problem (like reconstruction of interior structure) is being solved. In this work we propose a system for the measurement of the x-ray beam parameters based on usage of metallic (W, Au, Ni, etc.) tips. These geometrically simple objects can be fabricated quite precisely and admit their measurements (by a calibrated scanning or transmission electron microscopes) with high accuracy. Besides, they can be used for various values of the x-ray wavelength and intensity because of the presence of regions with different thickness (diameters).
We created mathematical model of the absorbed and fluorescent signal formation. An example of calculation is present on Fig.1 where intensity of transmitted signal from tungsten (W) needle is displayed for fine (infinitely thin) beam. Radius of needle(R) was 1.5 mk. An absorption signal for x-ray beam with spot α is given by

$$I(y_0) = \frac{1}{\sqrt{2\pi}R}\int dy \exp(-2\mu\sqrt{R^2-(y-y_c)^2} - \frac{(y-y_c)^2}{2\alpha^2}) \quad , \qquad (1)$$

where μ is the absorption coefficient, y_c is the position of the needle center. To find beam parameters we define functional Φ which describes difference between theoretical signal I_{th} and a measured one I_{exp}. $\Phi(\alpha, R, \mu, y_c) = \int dy_0 \, (I_{th} - I_{exp})^2$
Then we find such values of α, R, μ and y_c that functional Φ is minimal. The results of a calculation for Ni-Cr wire of 95μm diameter are shown on the Fig.2.

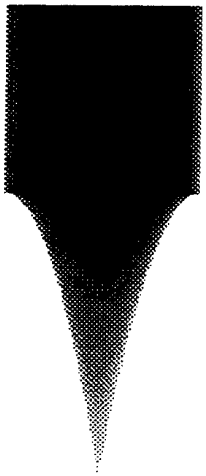

Fig.1

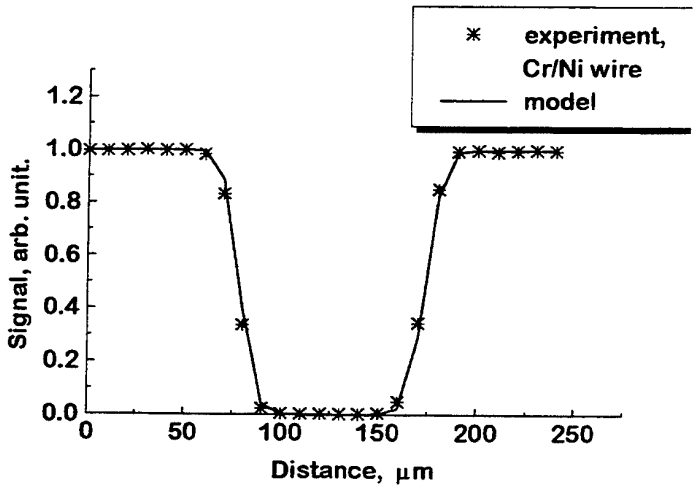

Fig.2

THEORY OF X-RAY SCATTERING BY ROUGH SURFACES WITHOUT DISTORTED WAVE APPROXIMATION

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In this paper we discuss the theory of x-ray scattering by rough surfaces and interfaces in layered materials and multilayered structures. The most of the theories of the x-ray scattering are based on the different variants of the distorted wave approximation. In this case the dielectric permittivity is averaged firstly over the random functions describing the interfaces and then the difference between the real and averaged dielectric permittivities is considered as a small perturbation.

Here we propose the new approach to the development of the theory of x-ray scattering. We expand the incident, reflected, scattered and transmitted waves into the Fourier series. The amplitudes of the waves in this expansion are determined by the boundary conditions at the rough surface. The specific feature of this approach is that the energy and energy flux conservation laws are fulfilled. For example, in the case of transparent materials the integral energy of the reflected, scattered and transmitted waves is equal to the energy of the incident wave. In the case of the multilayered structures we solve the boundary problem for the separate period and then we use the recurrence procedure to determine the integral scattered field. In general case the final equations are complex to be solved analytically but the computer algorithms can be easily constructed.

The proposed spectral approach is most effective for the analyses of scattering by regular surface and interface profile. As an example we considere the scattering due to excitation of the acoustic surface waves. For the case of the periodic profile of the interfaces, the solution can be obtained often in the analytic form.

Let σ is a root-mean square height of roughness, and k_z and q_z are the transversal components of the wave vector for the incident and scattered waves. When the condition $\gamma = (k_z + q_z)\ \sigma << 1$ holds we can use the perturbation theory with the small parameter γ. In this case we can get the solution for the scattered field in the analytic form. The specific feature of such a perturbation theory is that the effects of the dynamic diffraction are accounted both for the transmitted and scattered fields.

MEASUREMENT OF ELASTIC SHEAR STRAIN BY WHITE BEAM SR TOPOGRAPHY

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An external tensile stress applied to an elastically anisotropic cubic material along a direction different from cubic axis can cause a shear deformation. The shear strain is proportional to the anisotropy factor $\delta = s_{11} - s_{12} - \frac{1}{2}s_{44}$, where s_{11}, s_{12} and s_{44} are elastic compliances, and to the applied stress. For Fe - 4at%Si alloy $\delta = 8 \cdot 10^{-3}$ GPa^{-1} and shear strain is of the order of 10^{-4}. It is therefore very difficult to measure it on single crystals. In bicrystals, however, the shear strain can result in a mutual tilt of some planes at the grain boundary (GB), which is measurable by X-ray diffraction. The aim of this work is to compare the experimental results with the theoretical ones.

A $\Sigma 3$ bicrystal with the symmetrical GB ($x_2 = 0$) parallel to $\{112\}_{A,B}$ plane (A,B denotes the two grains) was chosen for the experiments. The external stress σ_{33} was parallel to the $<152>_{A,B}$ direction common for both grains . The compatibility condition $\varepsilon_{11}^{A} = \varepsilon_{11}^{B}$, $\varepsilon_{33}^{A} = \varepsilon_{33}^{B}$, $\varepsilon_{13}^{A} = \varepsilon_{13}^{B}$ [1] was fulfilled without any additional stresses. The shear component $\varepsilon_{12}^{A} = -\varepsilon_{12}^{B}$ results in the tilt $4\varepsilon_{12}$ of the plane $\{201\}_{A,B}$ ($x_1 = 0$). The specimen was observed under applied tensile stress by reflection topography using white beam SR at LURE (Orsay, France) [2].

A tilt of the diffracting $\{201\}_{A,B}$ planes was seen in the topographs as a gap or overlap of the images of A and B grains. The tilt increased with the applied stress and vanished after the stress relaxation. The amount of the tilt calculated from the overlap of the images was 30 - 40 secs of arc (estimated precision 8 secs of arc) and agreed with the values given by the theory of elasticity.

These results show that the small elastic shear strain can be measured using suitable bicrystals and taking advantage of the high sensitivity of the white beam SR topography to the tilt of diffracting planes. This method offers a possibility of testing the slight elastic anisotropy of cubic materials.

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[2] M Polcarová, A George, J Brádler, A Jacques and J Gemperlová, 2nd European Symposium 'X-Ray Topography and High Resolution Diffraction', Berlin 1994, p.11.

Surfactant mediated growth of Ge on Si(001) substrate:
a Normal Incidence X-ray Standing Wave study

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The growth of thick, low-defect, lattice mismatched films of Germanium on Si(001) substrate is an essential requirement for optoelectronic device applications. The mismatch in lattice constants give rise respectively to large strains in the overlayer which over a certain thickness can be relieved as dislocations, crystal defects or islands formation. The action of an appropriate third species (Sb in our case) can act as surfactant, lowering the energy of solid-vacuum interface, floating at the growth front without being incorporated and preventing from intermixing. In spite of its fundamental importance, the behaviour of Ge atoms at Ge/Si(001) interfaces with or without the presence of a surfactant is up to now an open question.

The X-ray Standing Wave (XSW) technique is a powerful tool to investigate interfaces and thin films (few monolayers): in fact, the simultaneous study of several reflections can give unequivocal information on the occupation sites of the adsorbate.

We have investigated the Ge absorption process at Si(001) surface as a function of annealing temperature and of surfactant action. Normal Incicdence XSW measurements have been performed at the 6.3 beam line of the Daresbury synchrotron radiation source. The samples have been grown and characterized by LEED and Auger spectroscopy in-situ (base pressure less than $1x10^{-10}$ torr). The Si, Ge and Sb photoelectron yields have been recorded as a function of incidence energy for (004), (022), (0$\underline{2}$2) and (111) substrate reflections.

Analysis of the experimental data gives evidence of important modifications of the Ge atomic behaviour in the presence of surfactant action of Antimony.

INTERFACE ROUGHNESS IN Ge_xSi_{1-x}/Si STRAINED-LAYER SUPERLATTICES

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ABSTRACT

Recently there has been considerable interests in the properties of epitaxial grown strained-layer superlattices (SLS's) of Ge_xSi_{1-x}/Si due to their potentially important applications in various optoelectronic and microelectronic devices. Since the quality of interfaces can greatly influence the strain and electron transport in thin-layer heterostructures, it is necessary to achieve a detailed knowledge of the interface microstructures. On the other hand, it has been recognized that the interface roughness exponent can spatially characterize dynamical scaling behavior of the growth of surface and interface. Although considerable theoretical schemes have been developed to determine the values of this exponent for different growth modes, there have been few experimental investigations for comparison, especially for the Ge_xSi_{1-x}/Si semiconductor superlattices.

In the present paper the interface roughness and scaling exponent of the Ge_xSi_{1-x}/Si SLS's grown by molecular beam epitaxy (MBE), were examined by x-ray reflectivity and diffuse scattering methods. Seven samples with different thickness of the superlattice periods and/or grown at different temperature of substrates were prepared and measured. With a goal of achieving sharp interfaces, a recent surfactant approach has succeeded impressing the segregation so that we also compared the interface morphology between the samples with and without surfactant. The reflectivity and diffuse scattering experiments were performed at the diffuse scattering station of 4W1C beam line of the Beijing Synchrotron Radiation Facilities (BSRF). The specular reflection was calculated by the recursive application of the optics theory-matrix method. For treating the non-specular diffuse scattering from interfaces, the distorted-wave Born approximation was accepted.

We have found that the interface roughness and scaling exponent vary with growth parameters and exhibit complexity. At same conditions the root-mean-square (rms) roughness (σ) increase, while the roughness exponent h and lateral correlation scales (ξ) decrease, with increasing the thickness of the Ge_xSi_{1-x} alloy layers in the superlattice periods. The incorporation of surfactant antimony can efficiently retard interface widening and smooth the interface. There must exists a appropriate range of temperature of substrates to achieve the SLS's with high quality otherwise over which will result in interdiffusion across the interfaces. We relate our experimental measurements of the interface roughness and scaling exponent to the dynamical character of the MBE growth and our results agree with the prediction concerning the roughness exponent by the Langevin equations and the Kardar-Parisi-Zhang (KPZ) equations. The roles of the surfactant to the interface morphology are also discussed.

It has been proved in this paper that the x-ray reflectivity combined with the diffuse scattering methods can provide the interface structures of superlattices with high resolution and accuracy. A simple approach, for distinguishing between the interdiffusion within bilayers and the true self-affine interface roughness, is also proposed.

DOUBLE-CRYSTAL X-RAY DIFFRACTOMETRY STUDIES OF SILICON GERMANIUM LAYERS ON SILICON SUBSTRATE

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The growth of $Si_{1-x}Ge_x$ alloy layers on silicon substrates opens new possibilities to modify the electronic band gap width and by this to develop high-frequency electronic circuits. But there are some difficulties to produce pseudo-morphic structures because of the large lattice mismatch between germanium and silicon (4 %) causing strained regions. Therefore, during the thermal treatment the crystal lattice can relaxe by growing misfit dislocations. The thickness and the depth (below the surface) of the layers and their strain can be studied with a high accuracy by high-resolution X-ray diffraction. Here results are reported from studies of layers produced by ion implantation and chemical vapour deposition (CVD), respectively.
In the first case isovalent impurities as Ge and C were implanted into the (001) silicon surface with ion energies in the MeV range, e.g. for Ge ions with an energy of 4 MeV the mean projected ion range is 2.74 µm. The lattice strain induced by the implantation was measured by the changes of the lattice plane distances. By rapid thermal annealing (30 seconds at 700 °C, 1000 °C, 1150 °C, and 1300 °C, respectively) the value of $\Delta d/d$ is reduced from $3 \cdot 10^{-3}$ to $2 \cdot 10^{-5}$. In this case the layer structure shows a small strain value and a large layer thickness. In Fig.1 measured double-crystal rocking curves are shown for the Ge implanted samples, and Fig.2 gives the measured maximum strain for Ge and C implanted samples after different thermal treatment.
Secondly layer structures with thicknesses of some 10 nm are produced by chemical vapour deposition of $Si_{1-x}Ge_x$ on the (001) surface of silicon. Here the germanium content varies between x=0 and 0.3 with a constant or a graded profile. Contrary to the implanted samples the CVD layer structure has high strain values and small layer thicknesses. The transition from the pseudo-morphic to the relaxed layer after thermal treatment was studied using (004), (224), (113) reflections.

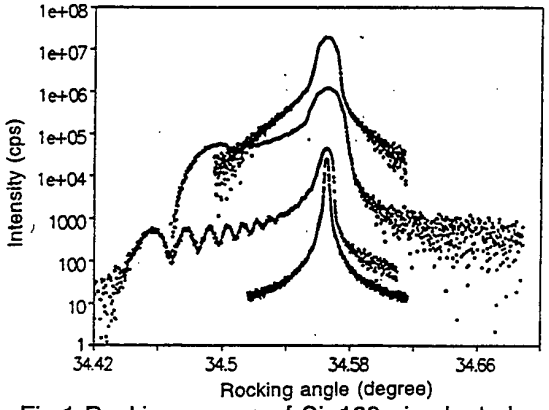

Fig.1 Rocking curves of Si<100> implanted by 4 MeV Ge ions $5 \cdot 10^{15}$ cm^{-2}. From bottom the instrumental curve and the rocking curves are shown in the as-implanted state and after annealing at 700 °C and 1000 °C, respectively. The curves are shifted along the intensity axis.

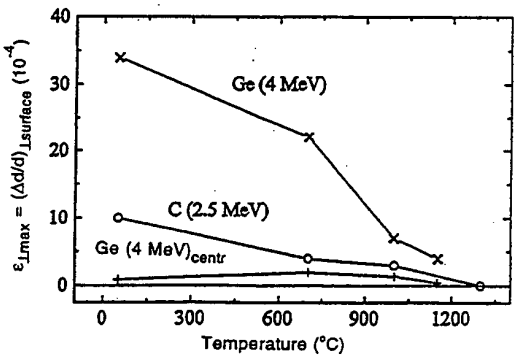

Fig.2 Maximum strain in the crystal lattice after implantation ($5 \cdot 10^{15}$ cm^{-2}) of Ge or C and rapid thermal annealing. Ge_{centr} means the absolute strain value deduced from the "substrate" peak.

CONTRIBUTION OF INTERNAL STRESSES TO SLIP TRANSITION ACROSS GRAIN BOUNDARIES

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The interaction of slip dislocations with grain boundaries (GBs) in plastically deformed Fe-4at%Si bicrystals with special low Σ GBs was investigated by white beam SR topography [1]. After the yield point was reached either isolated slip bands propagated from the edges of the specimen and stopped at the GB, or a group of homogeneously distributed slip bands developed relatively soon while the rest of specimen remained practically undeformed. The slip transmission across GBs cannot be fully explained by the geometric relations of the slip systems in the two grains. It was therefore suggested that stresses exerted by the slip bands in the first deformed grain and the local structure of the GB may significantly affect the transmission. Calculations of stress distribution near the GB were performed to explain the slip transfer over the GB for both cases observed in the topographs.

Elastic strain at the head of a slip band can be approximated by a macrodislocation with Burgers vector (BV) ranging from tens to hundreds of units of BV situated at the vicinity of the GB [2]. Stresses due to the macrodislocation are calculated.

The group of slip bands produces a plastic deformation. We suppose that the whole grain is plastically deformed. The grains are aproximated by elastic anisotropic half-spaces. Incompatibility due to homogeneous plastic deformation in one grain is accomodated by elastic strains [3].

The calculated additional stresses must be considered together with the applied stress to predict the slip system in the second grain. The maximum resolved shear stress (MRSS) and MRSS plane have been calculated for all possible BVs. It has been found that in all considered cases the slip continues on the primary slip system predicted by the applied stress in the undeformed grain even if the secondary slip system would be more favorable from the geometric point of view and its MRSS calculated for an applied stress is comparable to that one of the primary slip system. According to our observation direct dislocation transmission is not very probable. More probably the internal stresses are decisive for new dislocation sources to arise at the vicinity of GB.

The comparison of experimental observation and theoretical results will be presented.

[1] M Polcarová, J Brádler, A Jacques, A George and J Gemperlová, 2nd European Symposium 'X-Top 94', Berlin 1994, p.11.
[2] M Polcarová and J Brádler 1988, J.Appl.Cryst. 21,169-175.
[3] J Gemperlová, V Paidar and F Kroupa, 1989, Czech J. Phys. B39, 427-446.

X-ray Specular Reflectivity and Grazing Incidence X-ray Diffraction of new Langmuir-Blodgett multilayers

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In this work we report on a structural study performed by specular reflectivity, diffuse scattering and grazing angle x-ray diffraction measurements on new Langmuir Blodgett multilayers of a perylene-3,4,9,10-tetracarboxyldiimide derivative (PTCDI-Opent) and of a long chain-ammonium salt of 12-phosphomolybdate ($PMo_{12}O_{40}^{3-}$) deposited on (111)-oriented Si substrates. These new organic materials are promising candidates for the fabrication of electroluminescent devices or application in xerographic technology (PTCDI-Opent) and for the development of new electrochromic and electrocatalitic devices ($PMo_{12}O_{40}^{3-}$).

The periodic arrangement of the multilayers in the growth direction as well as the lateral crystalline in-plane ordering is investigated. Our results indicate a quite good structural quality of these complex organic molecules. Precisely, very well defined Kiessig fringes are present in the specular reflectivity spectra implying a uniform thickness of both the investigated structures. Moreover, both films are monophasic systems as determined by the existence of a single periodicity in the specular reflectivity spectra. The results of the lateral structural arrangement of the mulyilayers show a configuration in domains (70-140 Å) and a fairly good structural ordering within the domains. Thus, the diffuse scattering measurements of these multilayers is explained by small-angle scattering of the domains rather than interface roughness. Finally, from these investigations we can conclude that the structural quality of these new organic structures is comparable to multilayers from fatty acids and related salts.

XRPD APPLICATION FOR LASER-TREATED SURFACE OF FE-BASED ALLOYS STUDY

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Laser treatment to improve upon the physical and mechanical characteristics of alloys. The surface of laser-treated specimens of Fe–Ni alloys were investigated using X-ray powder diffraction and scanning electron microscopy methods and energy- as well as wave-dispersive X-ray spectral analysis. As a result it was found that microstructure of alloys was reduced to fragments by laser pulse treatment. In initial specimens of the alloys globular graphite surrounded by austenite (f.c.c.) phase was observed. But in laser quenching zone spherical carbides, needle-shaped α-martensite (b.c.c.) and residual austenite contained.

X-ray study of laser-treated thin surface layers showed irregular carbon and other alloying elements distribution throughout the treated layer. This fact is corroborated by nonmonotonous dependence of γ-solid solution lattice parameter deep into treated layer. In Fe–Ni steels containing excessive carbon as graphite and carbide phases laser melting of surface rised carbon solubility in γ-solid solution over equilibrium concentration value. In contrast, laser treatment of low-carbon steels decreased austenite carbon due to its burning content took place at the depth of over 60 μm.

Laser treatment of Fe–Mn–C alloy increased ϵ-martensite (h.c.p.) value due to M_S temperature shift of $\gamma \rightarrow \epsilon$ transition as result of austenite decarbonization.

HIGH RESOLUTION X-RAY DIFFRACTION STUDY OF POLISHING DAMAGE IN III-V SEMICONDUCTORS

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High resolution x-ray diffraction (HRXRD) has been performed on standard commercial single crystals of GaAs and InP which have been polished by various techniques. Chemical techniques include polishing with bromine methanol solution, hydrogen peroxide/ammonia solution, and sodium hydrochloride solution with alumina powder. Sections of one GaAs crystal were polished in the above ways, to make the conclusions independent of the substrate. The InP crystals were produced by various companies, each with their own polishing techniques, to show the applicability of this method as a means of substrate screening. A BEDE D^3 diffractometer was used to record triple axis reciprocal space maps, to allow tilt and strain contributions to be resolved. Use of symmetric and asymmetric reflections enabled the depth penetration to be varied and the in-plane and out of plane components of strain to be separated.

The FWHM and FW1/50M have been extracted from the maps, and compared, in both the q_y direction (corresponding to lattice tilts) and in the q_z direction (corresponding to changes in lattice parameter). It has been noted that for 004 symmetric reflections the lattice strain does not vary with sample preparation, but the tilt component to the rocking curve varies greatly. In the 224 asymmetric scans, where only approximately the top 1μm contributes to the rocking curve, the strain present is again virtually independent of the polishing technique. The tilt contribution is seen to vary, but less substantially than in the 004 scans. This implies that polishing principally affects the region of crystal well below the very top surface, and that refinement of polishing technique serves to remove tilts from the crystal, but does not affect the crystal strains. HRXRD has thus been shown to provide a quantitative comparison of polishing standards in III-V semiconductor wafers.

POLARIZATION EFFECT OF X-RAY PHOTOELECTRONS IN INVESTIGATION OF CRYSTAL SURFACE LAYERS.

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The external photoeffect is widely used in the method of X-Ray standing waves (XRSW) for studying the structure of thin surface single crystal layers. Energy analysis of the photoelectrons emitted from the crystal allows one to obtain information on the structure of layers tenths to several hundreds micron thick. The knowledge of the function $P(Z,\Delta E)$ which describes the probability of photoelectron escape from the depth Z with energy E is necessary for correct interpretation of experimental XRSW data from distorted crystals. Until recently, this analysis has been carried out under assumption that the angular distribution of the generated photoelectrons is isotropic.

This communication reports the results of the investigation of the influence of angular symmetry of the excited atomic state on the yield of the low-loss photoelectrons. It is shown that the polarization effect (PE) of photoelectrons can only be neglected in the case when the layer thickness exceeds the isotropization length. The experiments have shown that extreme values of PE, i.e. the ratio of photoelectron yield when $\mathbf{n} \parallel \sigma$ to photoelectron yield when $\mathbf{n} \perp \sigma$, are for the s-state 93%, p-state - 57% and for d-state - 31% (here n - unit vector in the direction of registration of photoelectrons, σ - polarization vector). PE has been investigated in the case of partially polarized X-Ray radiation. The experimental results are in a good agreement with theoretical models of photoelectron generation and transport in solids.

The influence of PE on the depth of escape of Si K-electrons and the accuracy of evaluation of the disturb layer depth in XRSW technique were investigated. The perfect Si(111) crystal with natural oxide layer of $L=20\div25$ A thickness on the surface was under investigation. It is shown that the change of polarization of X-Ray beam leads to the change of the effective escape depth of the photoelectrons on the tens of percents. Neglecting this obstacle may lead to wrong results. In the described experiment divergence from the true value of the disturb layer depth will be near 60%.As it is known in XRSW method more or less polarized radiation is used. For the aims of increasing XRSW method accuracy investigations of the dependency of $P(Z,\Delta E)$ from the polarization degree of X-Ray beam are actual.

Original multi functional goniometric mechanism with vacuum chamber are described.

Investigation of vertically structurized surfaces gratings by means of X-ray grazing incidence diffraction

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The advantage of the X-ray grazing incidence diffraction (GID) method is the opportunity to reduce the penetration depth to some nanometers below the sample surface. Thus the technique is most sensitive for the investigation of surface gratings.
In this work we present first results obtained from vertically structurized surface gratings. A sequence of 25 nm $Ga_{1-x}Al_xAs$ ($x\approx0.4$), 5 nm GaAs, again 25 nm $Ga_{1-x}Al_xAs$ ($x\approx0.4$) and a 5 nm GaAs capping layer has been grown by MBE. After organic cleaning and oxide removal the sample were spin coated with a 140nm thick layer of negative tone photoresist. The holographically exposed resist was used as an etch mask for the wet chemical etch process. A CH_3OH: H_3PO_4: H_2O_2 (10:1:1) solution (etch rate 180 nm/min at 18°C) was used to etch down through the GaAlAs-layer and create deep etched free-standing wires. The actual wire widths was determined by measuring the geometrical size of the etched nanostructures by high-resolution field-emission-scanning electron microscopy. The lateral period obtained was about 500 nm. The valley to wire ratio was changed between 7:1 and 3:1. Because of the relative strong damage of the wire sidewalls caused by the etching process we did not found any satellite peaks using high-resolution X-ray diffraction.
The GID measurements were performed at the W1 beamline of the Hamburger Synchrotronstrahlungslabor (HASYLAB) using a wavelength of 0.14 nm. The [001] oriented surface was illuminated by a well collimated incident beam under an angle of incidence lower than the critical angle of total external reflection in order to realize an effective penetration depth of about 5 nm below the surface. Thus the diffraction signal of the used (220) and (-220) reflection is mainly due to the surface grating whereas the scattering contribution of the unstructurized GaAs substrate can be neglected. Because of the relative low angular resolution of the used position-sensitive detector in the plane of diffraction which was aligned perpendicular to the sample surface we mainly performed "omega-scans" across the respective in-plane Bragg positions. We found intensive oszillations of the diffraction signal if the diffraction vector was directed perpendicular to the grating. The angular distance between the oszillations measures the grating period. The oszillations themselves appear over-modulated on a twin peak which allows to determine the shape of the wires. If the diffraction vector was directed parallel to the grating we found low intensive short-frequently oszillations superimposed on the single Bragg-peak which is explained by the scattering at the density modulated lattice. The measured intensity distribution will be explained in terms of the extended kinematical theory [1] taking carefully into account the resolution function of experiment.

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WHITE BEAM SR TOPOGRAPHY OF PLASTICALLY DEFORMED Fe - 4at%Si BICRYSTALS

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The plastic behaviour of metals is greatly affected by grain boundaries (GBs). Interaction of slip dislocations with GBs can be best studied on bicrystals where the GBs are well defined. The slip transmission depends on the slip geometry, i.e. on the directions of Burger vectors and slip planes in both grains with respect to the GB. In addition the stresses exerted by dislocation pile-ups in one grain are important for the slip formation in the second grain. The aim of this work is to compare results on specimens deformed by tension (T) and by compression (C).

The GBs, slip bands and other defects were observed by reflection topography using white beam synchrotron radiation at LURE (Orsay, France) [1]. Three types of specimens with different geometry of slip systems were chosen for both T and C experiments. The T specimens were examined *in situ* under applied external stress. The specimen state, as seen on the topographs, has not changed after the stress relaxation. The results can be therefore compared with those obtained after the deformation on the C specimens.

The main features different in T and C specimens are:

- Concentration of deformation in one or few limited regions, frequently seen in T specimens, was not observed in C specimens. This can be explained by reduced (enlarged) specimen cross-section at the slip band followed by increased (decreased) local stress in the T (C) specimens. This effect can be observed in single crystals as well.
- In T specimens the slip transfer over the GBs happens either by individual slip bands or, more frequently, by groups of them, while in C specimens only individual slip bands appear behind the GB and the stresses due to dislocation pile-ups are lower. This effect can be explained by different thickness of the specimens. In the thicker C specimens a higher number of defects at the cross-section of a slip band with the GB can result in higher probability of nucleation of new slip bands.

It can be concluded that the geometry of the slip is not the dominant factor for the slip transfer over the GB. The GB forms a strong barrier for the slip transmission also in the case of the most favourable geometry, i.e. both Burgers vectors and slip planes parallel in the two grains. It seems that the stresses exerted by dislocations accumulated at the heads of the slip bands at the GB together with the local structure of the GB are most important for the slip transfer.

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INTERFERENCE PHENOMENA IN X-RAY SCATTERING FROM MULTILAYER NANOSTRUCTURES

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We report the results of the experimental and theoretical study on the x-ray diffuse scattering by multilayer nanostructures. We have experimentally observed a number of phenomena which are due to the effects of the dynamic diffraction. The theory of the diffuse scattering based on the combination of the two-wave dynamic diffraction and distorted wave approximation [1] is developed. We have received the expression for the intensity angular spectra of the diffuse scattering in the analytic form, which takes into account the appearance of the maxima of the resonant diffuse scattering and describes the dependency of the scattering profile on the statistical characteristics of the interface roughness (rms height of roughness, longitudinal and transversal correlation lengths).

In our experiments we have used the different x-ray mirrors with the periods $d=a+b$ from one up to twenty nanometers consisting of the different pairs of materials, and with the different ratios of the layers thickness $\beta=a/d=0.1\text{-}0.5$. It is shown that the effects of the dynamic diffraction associated with both transmitted and scattered waves lead to the strong dependency of the scattered wave intensity on the angle of incidence near the region of Bragg reflection [2]. The effects of the forbidden reflection release, the rise of the background intensity, and the appearance of the additional maxima of the diffuse scattering have been observed experimentally. In the region of Bragg diffraction, the angular profile of scattered radiation intensity depends not only on the value of the parameter β, but on the statistical parameters of the layer roughness [3].

We have developed the optimized experimental procedure for reconstruction of the statistical characteristics of the interface roughness from the angular spectra of the scattered radiation intensity.

The good agreement of the theoretical and experimental angular spectra of the diffuse scattering enables us to determine with the help of computer fitting the following statistical parameters of the investigated mirrors: the rms height of roughness, longitudinal and transversal correlation lengths.

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Modelling imperfections of epitaxial heterostructures by means of x-ray diffraction analysis

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High-resolution double-crystal x-ray diffractometry provides accurate information on the structural configuration of multilayered semiconductor heterostructures. Since the information on phase shifts of x-ray wave fields is lost in the experimental diffraction curves, the measured diffraction curves have to be analyzed by the dynamical diffraction theory. Deviations between measured and simulated data indicate imperfections within layers and at interfaces with a resolution down to an atomic scale. A modelling of the realized heterostructure is usually acquired by means of the "best fit" of the measured curves, which is satisfactory to support the improvement of material growth. However, this fit is mostly performed by a visual comparison between measured and simulated data in an iterative and highly subjective manner. The aim of this work is to present a method of modelling imperfections in semiconductor heterostructures. The key steps of this method are (i) the sensitivity analysis of experimental diffraction curves with respect to imperfections, and (ii) the development of objective error criteria.

The applicability of this method is demonstrated using highly strained $[(InAs/GaAs)_n/InGaAs]_m$ superlattices grown by molecular beam epitaxy (MBE) on InP substrates. Symmetric (004) x-ray diffraction measurements are performed using a STADI P x-ray diffractometer with a double-crystal monochromator. While the individual layer thickness within the superlattice can be directly calculated [1], the interface fluctuations are subject to a modelling process. Intermixing at the InAs/GaAs interfaces results in a reduction of the lattice strain and a sensitivity analysis revealed that the overall intensity of the satellites is significantly affected. Hence the satellites intensity or rather the ratio of the different satellites intensities are taken as an error function for the calculation of the agreement between the measured and simulated diffraction curves. The final result of this modelling process is a quantitative and objective determination of the intermixing at InAs/GaAs interfaces in relation to the MBE growth parameters which can be transferred to any other laboratory.

In addition, sensitivity criteria will be presented for both fluctuations within a layer and at interfaces, in order to demonstrate that our approach may be extended to other structural imperfections in multilayered heterostructures.

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Reduction of misfit dislocation density in strained $In_xGa_{(1-x)}As$ heterostructure via growth on patterned GaAs (001) substrates

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The <110> oriented misfit dislocation density reduction in the growth of $In_xGa_{(1-x)}As$ (x=0.06) heterostructure on GaAs (001) substrate patterned with circular mesas has been observed with x-ray double crystal topography. It was found that the most of threading dislocations locating at the mesa valleys cannot cross the mesas, while the threading dislocations locating at the mesas can easily glide to the valleys. No misfit dislocation is observed terminating on the top of the mesas. The experimental results obtained here are explained by the resistant effect of mesas from the point view of the elastic strain energy of patterend region.

VERTICALLY CORRELATED ROUGHNESS WITH LARGE LATERAL LENGTH SCALE IN STRAINED InGaAsP/InGaAsP SUPERLATTICES

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In the last few years, complex heterostructures based on strain controlled InGaAsP material have been widely used to design optoelectronic devices with improved performances [1]. On the other hand it has been recognized that surfaces and interfaces under stress can exhibit spontaneous roughening which may hamper the performances of the heterostructures [2]. We have investigated device quality strained InGaAsP/InGaAsP superlattices, grown by organo-metallic vapor phase epitaxy, by means of triple axis X-ray diffraction in different azimuthal geometries. The observation in reciprocal-space maps of a well defined lateral periodic modulation oriented along the miscut direction of the substrate, is interpreted as the existence of a vertically correlated roughness. The lateral wavelength is in the micron range and corresponds to a much larger length scale than that of the average steps separation expected from the substract miscut. Atomic-force microscopy measurements of the surface morphology of the superlattices corroborate the X-ray measurements and in addition suggest that the large lateral periodicity results from a modulation of the distance between steps . We will discuss the origin of these phenomena and stress that they are of different nature than the self-induced laterally modulated structures, with smaller characteristic wavelength and different orientation, already reported in strained III-V compounds [2]. In particular we will discuss, according to recent experimental and theoretical works on SiGe/Si compounds [3,4], the consistency of our observations and data with a roughening mechanism implying the formation of step bunches driven by strain relaxation at the substrate steps.

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GLANCING INCIDENCE X-RAY CHARACTERIZATION OF Nb/Pd AND Nb/CuMn MULTILAYERS

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In recent years, the study of periodic metallic multilayers in which one of the two constituent layers is a superconductor has attracted considerable interest, because these systems offer the possibility of creating new material structures with novel physical properties. Various combinations of elements, alloys and compounds have been investigated in order to tailor the superconducting properties of the whole structure. In this framework, the structural configuration and quality of the interfaces is of fundamental importance because it influences the phase of the superconductive wave function in the interface region and, hence, the coupling between the nearest superconductive layers.

In this work, we present a structural investigation of Nb/Pd and Nb/CuMn multilayers by using high- and low-angle x-ray diffraction measurements. All the samples were grown on Si(100) substrates by using dc-triode sputtering and employing a magnetically confined Ar plasma system for the deposition of Pd and CuMn, and a magnetron system for Nb, both placed in the same vacuum chamber. The superlattice period of the samples investigated here ranges between 27.4 nm and 21.5 nm, and each structure is made of 10 stacks.

The x-ray analysis has been performed with a single-axis diffractometer optimized for glancing angles measurements and by using CuKα radiation. High-angle diffraction measurements are performed in order to identify the phases in the constituent layers and to determine the preferred crystallographic orientation. The θ/2θ scans recorded in the angular range between 5° and 100° reveal that the Nb layer is oriented in the [110]-direction and the Pd, CuMn layers in the [111]-direction. Specular reflectivity measurements, reciprocal space maps with transverse and off-specular scans were performed in order to characterize the interfaces in the directions perpendicular and parallel to the surface. The experiments were compared with calculations by using the distorted-wave Born-approximation (DWBA). From the reciprocal space maps, the presence of diffuse intensity localized near the Bragg planes implies the existence of a correlated or partially correlated roughness between the interfaces. The specular scans reveal a distribution of the rms roughness along the growth direction from few angstroms near the substrate to about ten angstroms near the surface.

X-RAY DIFFUSE SCATTERING AS A PROBE FOR SURFACE STRUCTURES OF SILICON WAFERS

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Chemically clean and physically smooth silicon surfaces are an important prerequisite to produce perfect microelectronic structures. Teichert et al. [1] showed that x-ray scattering from rough surfaces enables to complement data about the surface morphology obtained by other methods like atomic force microscopy.

The silicon wafers under investigation are unpolished samples and samples that have been cleaned by alternative chemomechanical procedures. X-ray specular reflectivity (XSR) and x-ray diffuse scattering (XDS) from each silicon surface are measured to determine the surface roughness σ and the lateral correlation length ξ.

Using a laboratory x-ray diffractometer (λ = 0.154nm), a parabolic, graded multilayer mirror is inserted [2] to increase the incident flux of x-rays onto the sample surface. Additionally, the coherence length of x-rays parallel to the sample surface is enlarged. XDS scans are accomplished by so called ω scans in accordance with the instrumental settings of the diffractometer.

Due to the rotation of the sample around the surface normal (azimuthal angle ϕ), an anisotropic scattering from the surface is observed. This results in an asymmetrical diffuse component in XDS. The anisotropic scattering is not caused by surface wavyness or by wafer curvature. Azimuthal dependent scattering has already been observed in light scattering experiments by Domashev et al. [3]. It is explained by extended anisotropic structures like steps and terraces present on silicon surfaces. In addition to the asymmetry mentioned above, intensity changes of the specular reflectivity are dependent on the azimuthal angle ϕ. As a consequence, XSR measurements with a high angular resolution can provide different values of roughness dependent on the angle ϕ. The effect of anisotropic surface structures extending the range of surface roughness σ (0.05nm..2nm) has to be considered for the assessment of the surface quality of silicon wafers.

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COMPARISON BETWEEN DIFFERENT X-RAY DIFFRACTION METHODS TO EXTRACT STRAINS IN METALLIC MULTILAYERS

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It is well known that, in multilayers, the information on perpendicular lattice strains is hidden in the intensity of the superlattice peaks, whereas the positions of the diffraction lines are related to the superperiod. This calls for some refinement , and a number of kinematic modelling schemes have been given in the literature. On the other hand, the in plane directions, as measured by x-ray diffraction in the grazing incidence or the transmission modes, do not involve any superstructure; thus they can lead straightforwardly to in plane distances. From the perpendicular (deduced from modelling) and the in-plane interreticular distances, elastic strains may be deduced through a standard elasticity analysis, assuming a biaxial strain state ($\varepsilon_\perp$ / $\varepsilon_{//}$ method).

In order to get the full strain tensor, it is usual to perform a "$\mathrm{Sin}^2\psi$ analysis" (ψ is the angle between the lattice plane normal and the sample surface normal). This is a standard way to extract stresses in a variety of materials. This method relies on the measurement of asymmetric Bragg peaks which may contain superlattice reflections.

In this communication we will present a detailed comparison between the $\mathrm{Sin}^2\psi$ technique, i.e. lattice strain measurements from asymmetric Bragg peaks, and the $\varepsilon_\perp$ / $\varepsilon_{//}$ method. This study is performed on Au-Ni multilayers. One of the major conclusions is that the $\mathrm{Sin}^2\psi$ analysis is always valid but the measurement of lattice strain becomes more complicated when the layers are epitaxial. The measurement method is highly dependent on the crystal quality. Epitaxial multilayers call for $q_\perp$ scans modelling of the asymmetric reflections, whereas in polycrystalline samples the standard measurement of lattice strain remains valid.

The Images of Misfit Dislocations in Bragg-Case Synchrotron Section Topography

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The silicon epitaxial layers with a misfit factor 5×10^{-5} caused by doping of the substrate with boron were studied by means of back-reflection synchrotron section topography. The synchrotron experiments were realized using white radiation beam limited by 5 μm wide slit. The presently examined samples with the thickness 10 - 20 μm contained usually certain amount of misfit dislocation.

It was found that when the curvature of the sample was negligible the section pattern contained of two fringes corresponding respectively to the reflection from the surface of the layer and the surface of the substrate. In this case the misfit dislocations were revealed in the direct contrast most intense in the vicinity of the fringe due to the reflection from the substrate.

The observed pattern became significantly different when the sample had a certain curvature. In this case additional interference fringes were observed in wide area behind the main two fringes. The sequence of these interference fringes was dependent on the curvature of the sample and the reflection. It was also considerably different from that of bent substrate wafers with the similar curvature. As a consequence the images of misfit dislocations in the bent epitaxial layers also significantly changed. They became much more extended and contained many characteristic details.

The contrast of interference fringes in epitaxial layer and of misfit dislocations was reproduced with a reasonable accuracy both in the case of flat and bent crystals using numerical integration of the Takagi-Taupin equations. The present results confirmed former identification of misfit dislocations type as mixed 60°, obtained using numerical simulation of the double-crystal images.

WAVE-OPTICAL DESCRIPTION OF X-RAY PHASE CONTRAST IMAGES OF WEAKLY ABSORBING NON-CRYSTALLINE OBJECTS WITH A HIGH-RESOLUTION DIFFRACTOMETRY

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It is known that X-ray transmission radiography of non-crystalline objects with extremely small absorption gradient is practically impossible by means of traditional contact radiography and tomography methods. However the problem of the image obtaining of such objects is very important in medicine, biology and materials science.

The main idea of the X-ray contrast images enhancing consists of a high-resolution angular registration of radiation transmitted through an object with the help of perfect crystal analyzer [1-4]. A phase gradient across a wavefront of transmitted wave, which appears due to the differences paths lengths and refractive indices $\delta = 1 - n(x, z)$, is the same as a change in the direction of wave propagation. Since the refractive indices of biological tissues are small, $\delta \sim 10^{-6}$, one needs an angular resolution of about 0.1 arc sec for observation of interference images.

The aims of the present work are: (1) wave-optical description of X-ray phase contrast with crystal-analyzer in Laue geometry, (2) determination of optimum experimental conditions for contrast enhancing (wave divergence after monochromator, crystal-analyzer thickness, reflection indices, deviation angle from exact Bragg position), and (3) comparison with the experimental images of different model objects (PMMA prism, kapron cylinder, and polymer capillary).

The complex amplitudes of the reflected and transmitted waves at the exit crystal analyzer surface are defined by the convolution of the Green function with the arbitrary phase-modulated wave field at the entrance surface. Analyzer positioned in Laue-geometry, which was suggested in [3], is more sensitive in comparison with one positioned in Bragg-geometry. It is connected with the phase accumulation effect in the crystals-analyzer with intermediate thickness $\mu t \sim 0.5 \div 1.5$, and with the presence of two complementary images in *R*- and *T*-beams. Wave interference manifests as a fine structure of black-and-white contrast especially in the images of small objects and boundaries with large phase gradient. Images of objects having dimensions much more exceeding extinction length, and far away from the sharp object edges can be calculated by the simple geometrical optic approximation. The numerical calculations of the model objects images are found to agree with the experiments.

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DETAILED STUDY OF THE MAIN FEATURES OF THE PROCESS OF ELASTIC STRAIN RELAXATION IN GAAS/ZNSE HETEROSTRUCTURES.

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HRXRD and TEM, which have extremely high sensitivity to crystal lattice perfection have been used for detail investigation of the main features of the process of elastic strain relaxation in ZnSe layers grown by MBE directly on nominally cut GaAs(001) substrates. Each investigated sample has been grown under the same perfect growth conditions with the thickness of ZnSe layers varying from 50 nm up to 1 μm. Various stages of the process of elastic strain relaxation, beginning from perfect elasticly strained structure up to nearby completely relaxed, have been studied.

The process of elastic strain relaxation is accompanied by generation of different structural defects such as clusters of point defects, stacking faults, dislocation loops and misfit dislocations. Every stage of relaxation can be characterized by the main type(s) of structural defects, which strongly influence by definite way on crystal lattice perfection of heterostructures. Investigation of these structures is rather difficult problem and needs correct interpretation.

Combined application of HRXRD and TEM for investigation of the process of elastic strain relaxation allowed one to get the main correlations between type of structural defects and reciprocal space distribution of diffracted x-ray intensity. It allows one to simplify the investigation procedure and hence the interpretation of received results.

TO THE PROBLEM OF INFLUENCE OF THE COMBINED STRUCTURE DISTORTIONS OF VARIOUS LENGTHS ON THE DYNAMICAL SCATTERING OF X-RAYS

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The most complicated case of X-rays scattering is realized in crystals containing simultaneously both macroscopically distributed elastic strains and localized distortion fields due to structure defects. This difficult problem is not solved till now not only theoretically but by experimental methods too. It is of interest, for example, to study effect of short- and long extended distortions on the extinction length Λ_0 which is one of the important characteristics of scattering. Influence of the two independent (short- and long wave distortions as comparing with the parameter Λ_0 for a perfect crystal) ultrasonic vibrations (USV) or periodic static waves of displacements on the Λ_0 in a perfect silicon crystal have been studied. In the first case the Λ_0 changes due to the USV were measured by analysis of the spatial intensity distribution of the Borrmann diffracted beam [1] when the sample was excited by the second modeling source of USV. In the second case the effect of the SW on the Pendellosung phenomenon in the thickness integral reflectivity dependence was modeled by solving of the corresponding equations. In both of the cases an increase of the Λ was shown for the short wave periodic displacements as comparing with Λ_0 and decrease of this value for the long wave distortions of a structure. The additive influence of the two independent vibrations on the Λ_0 was established at low level of deformations i.e. HW=0.1 where H and W are the diffraction vector for given reflection and an amplitude of vibrations respectively.

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PERIODIC DOMAIN INVERSION IN $LiNbO_3$ AND $KTiOPO_4$ *VIA* MULTIPLE-CRYSTAL AND SYNCHROTRON TOPOGRAPHY

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Compact blue-green light sources based on the frequency doubling or second-harmonic generation (SHG) of laser diodes are very attractive for applications such as biomedical instrumentation, high density optical data storage, printing, and colour displays. Compared with natural birefringent phase matching which is only available in limited range of anisotropic crystals, the quasi-phase-matching (QPM) technique, where phase matching is achieved by periodic modulation of the nonlinear coefficient along the direction of propagation, results in high nonlinear conversion efficiency and phase-matching of any desired wavelength within the transparency range of a crystal. QPM can be achieved most efficiently by introducing a periodic array of domains of alternating structural polarity or so-called periodic domain inversion (PDI) into a ferroelectric crystal. PDI has been optically demonstrated in the well-known crystals, $KTiOPO_4$ (KTP), $LiNbO_3$ (LN), and $LiTaO_3$. Since the future development of superior nonlinear optical media is likely to depend more on the utilization of tailoring techniques such as PDI than the development of brand new materials, it is of considerable importance to develop methods for examining PDI and gaining an insight into the domain-inversion process at the microstructural level.

Multiple-crystal multiple-reflection topography in high spatial resolution mode and synchrotron topography were combined to characterize periodically domain-inverted structures in LN and KTP. In LN periodically-poled using an electric field, fine linear contrast with a spacing which corresponded to the lateral dimension of the periodic pattern, was observed by choosing an appropriate diffraction mode to provide high spatial resolution. The observed contrast is shown to arise from strains generated at the unnatural domain walls forced to exist within the crystal. In KTP poled by electron beam direct writing, no strain was observed at the domain walls. However, the inversion domains in KTP have been clearly imaged in multiple-crystal X-ray topographs of carefully-chosen reflections with strong anomalous scattering, and weak dynamical contrast at the domain walls has been revealed *via* synchrotron white-beam topography. An interpretation of the origin of topographic contrast is given, and experimental results are examined within the context of structural models for domain-inversion in LN and KTP and found to be consistent with our structural analyses.

NEUTRON INELASTIC SCATTERING ON ULTRASONIC EXCITATIONS IN SILICON AND GRAPHITE SINGLE CRYSTALS

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The influence of the ultrasonic acoustic waves (AWs) on x-ray and neutron scattering in single crystals has been the subject of many investigations. The energy E of the AW phonons is very small ($E \approx 4\ 10^{-7}$ eV when the AW frequency f=100 MHz). Therefore the energy spectrum of the diffracted beam was not studied (as a rule).

The aim of this work was the studies of the neutron *inelastic* scattering on the bulky high-frequency ultrasonic excitation in single crystals. We applied the method of the neutron spin-echo and directly observed for the first time such scattering. We studied symmetric Bragg reflections (111) and (002) from Si and PG - graphite ($\Theta_B \approx 60°$, $\lambda \approx 0.54$nm, $\Delta\lambda/\lambda = 18\%$, beam collimator ~0.5°). The start intensities of the diffracted beam were ~100 imp/s and ~ 800 imp/s for the case of Si and PG samples. The transversal (TAW) and longitudinal (LAW) ultrasonic waves were excited by means of $LiNbO_3$ transducers. The wave vector **k** of the TAW was *perpendicular* to plane of scattering. The influence of the TAWs and LAWs on the diffraction was similar in our experiments. The final neutron polarization P(V,H) was measured as a function of the applied magnetic field H in the coils, voltage of the h.f. generator V and f. Fourier analysis of P(V,H) - intermediate function of scattering - allowed to find the intensities of the elastic I(0), one-phonon I(1) and double-phonons I(2) scattering.

Perfect Si crystal. We observed one-phonon scattering (LAW, 209 MHz), I(0) was almost independent from the AW amplitude W. The exponential decay of the polarization was observed for the case of strong excited AWs.

Bended Si crystal. In the *deformed* Si very small ultrasonic signal (f_s=49 MHz, V=6 mV, $W \sim 10^{-2}$ nm) *decreases* the intensity of the elastic scattering up to 30 %. This decreasing doesn't compensated by the I(1), I(2) scattering, so that the total intensity of the diffracted beam decreases up to 18 %. These results correspond to our previous theoretical model (one-phonon satellites are presented mainly in the *forward* scattered beam) and neutron and x-ray experiments.

Graphite. We observed strong inelastic scattering in PG single crystal excited by the LAW (f=30.5 MHz). The structure of the PG crystals are very far from perfect and effect of the AW on the *total* intensity is *absent*. Our results may be interpreted as a motion of an each mosaic block as a whole in the AW (W~0.06 nm).

It seems obviously that the neutron spin-echo method may be applied for the studies of the ultrasonic excitation in the wide class of samples. The energy analysis of the diffracted beam allows to measure the distribution of the AW amplitude not only in high-quality single crystals but in the ordinary crystals and even non-crystalline materials. The similar results may be obtained also (in principle) by means of Rayleigh Scattering of Mössbauer radiation with Synchrotron excitation of the Mössbauer nuclei.

X-RAY MULTILAYER MIRROR WITH WIDE ANGULAR AND SPECTRAL REGION AND ITS APPLICATION TO X-RAY MICROSCOPY

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Some branches of X-ray physics, such as focusing of X-ray beams, synchrotron experiments or X-ray microscopy need the mirrors with the angular reflection region of 0.5° and more for CuKα radiation. At this wavelength traditional X-ray mirrors, including conventional multilayer structures, provide the angular reflection region not more than 0.1°. As one of the possible solutions of this problem we propose a multilayer mirror with a varying period. In this work is shown, that a multilayer mirror prepared by magnetron sputtering process may have a reflection region of 0.4°-0.5° with a maximum reflectivity of 40-30% respectively (fig.1), if its periods are properly distributed by depth. Theoretical prediction based on Parratt iterative procedure is in excellent agreement with the experimental data (fig.1). The mirrors of this type have also a wide spectral range. As one of the applications an X-ray raster scanning microscope was constructed and successfully tested. An image of a copper grid with a period of 110μm is portraited at fig.2.

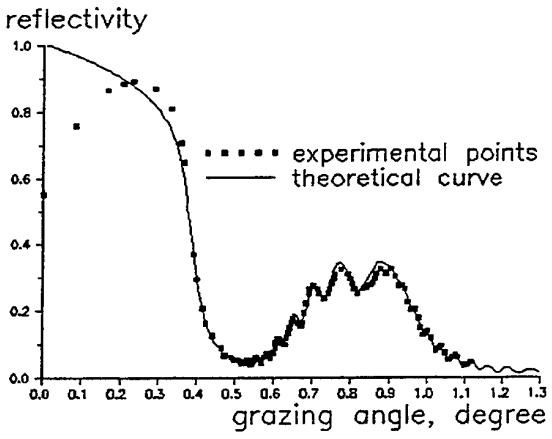

Fig.1. The reflection curve for W–Al multilayer mirror. 20 periods.

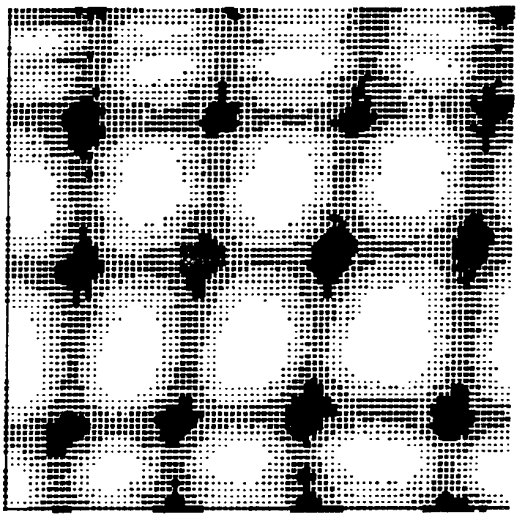

Fig.2. The image of a copper grid. Four brightness levels: 0.1, 0.2, 0.3 and 0.4

HIGH-RESOLUTION X-RAY DIFFRACTION OF SILICON AT LOW TEMPERATURES

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Since the early stage of high-angle double-crystal X-ray diffractometry (HADOX) experiment, anomalous behaviour of silicon has been found and investigated by means of variours techniques. The results of these experiments have shown that the lattice spacing of silicon is stress sensitive and, in general, depends on the thermal history at low temperatures; the temperature dependence of the lattice spacing d(T) is irreversible for cooling and heating. It is also shown that these phenomena may be related to the appearance of the negative thermal expansion below about 120 K. The purpose of the present experiment is to obtain more information on the anomalous behaviour of the lattice spacing of silicon by extending the temperature range of measurements down to 20 K, and by allowing several successive thermal cycles. The X-ray diffractometer used in the present experiment consists of a germanium quadruple-crystal monochromator(Philips PW1754) and a four-circle goniometer(Huber 511.1/424). Measurements of the lattice spacing of silicon were made by the Bond method on the 444 diffraction at Bragg angle $\theta \sim 79$ ° with Cu $K\alpha_1$ radiation. The results show that the lattice spacing d of (444) is not a one-to-one function of temperature, and has different values on cooling and heating. The characteristics of d(T) observed after repeating thermal cycles are as follows. (1)The difference between d values on cooling and those on heating decreases, and d(T) approaches a final curve. (2) While d values at temperatures above 120 K monotonically vary, those at lower temperatures show complicated behaviour. (3) Anomalies around 60 K become significant after the thermal cycles. The results suggest an existence of a new relaxation process at low temperatures for rearranging defects and host atoms in the silicon lattice .

Treatment of the heat-load associated contrast in Synchrotron Radiation Topography

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The third generation synchrotron radiation high-energy sources like the ESRF have interesting properties for diffraction topography [1]. The small source size associated with the natural collimation of the synchrotron radiation enhances both the spatial resolution and the sensitivity to weaker distortions. The higher intensity allows real-time experiments at shorter time scales.

The high photon flux of these new sources results in a large incoming beam power density (1-10 W/mm^2) on the first crystal (monochromator or sample). When a substantial part of the photons are absorbed the deposited energy may heavily modify the diffraction properties of the crystal because of the thermal expansion, hence affecting the topographs.

We present a model which explains the contrast associated to the thermal deformation of the sample. We consider the absorbed power, the mechanical, thermal, as well as the diffraction properties in order to get a simple rule for the duration (and/or filtering) of a topographic experiment in order to obtain minimized thermal effects corresponding to a maximum distortion $\delta\theta_{max}$. We show that it is possible to define a characteristic heat diffusion time t_D which can be compared with the exposure time t_{exp}. We already published the criterion for the stationary case $t_{exp} \gg t_D$ [1],

$$\frac{\delta\theta_{max}}{\omega_D} \simeq \frac{\beta\, P_{abs}\, l}{k\, \omega_D} < 1 \ ,$$

β being the thermal expansion coefficient, k the thermal conductivity, P_{abs} the absorbed power density, l the half beam vertical size and ω_D the Darwin width. A useful application of this criterion is the preliminary estimation of the thermal distortions of the first monochromator crystal, which usually has to operate for several hours under the beam.

Here we give a solution for the general transient case $t_{exp} \leq t_D$. We show that this solution can be reduced to the simple criterion

$$\frac{\delta\theta_{max}}{\omega_D} \simeq \sqrt{\frac{\alpha\, t_{exp}}{\pi}}\ \frac{\beta\, P_{abs}\, l}{k\, \omega_D} < 1 \ ,$$

α being the thermal diffusivity.

We show that good topographs can be obtained by using a filtering device or a stroboscopic beam shutter, which allows to cut the beam in short pulses in order to keep low the heat load, and consequently the distortion gradient, in the sample.

The use of these devices lead to experimental results which can be explained by the present criteria.

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INTEGRATED X-RAY SUBSTRUCTURE ANALYSIS OF PLASTICALLY DEFORMED BERYLLIUM SINGLE CRYSTALS

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Beryllium single crystals are characterized by a strongly anisotropic deformation behaviour caused by extremely different critical resolved shear stresses of the possible slip systems [1]. This results, for instance, in serious complications for the preparation of a neutron monochromator. Prismatic slip as the second order slip system was applied to produce a suitable substructure for that [2].

For an integrated analysis of the substructure of the crystals the X-ray double crystal technique is a suitable tool, using measurements of both:

- the radial intensity distributions I (2θ) and
- rocking curves (the azimuthal intensity distributions) I (ω)

of the reflections (0002), ($10\bar{1}0$) and ($11\bar{2}0$). Account has taken of the low absorption of X-rays in beryllium and the elastic anisotropy.

From the line broadening of the intensity distribution (2θ), which is caused by the local lattice strains of the irradiated specimen, the mean dislocation densities ρ can be estimated via Fourier analysis of the peak shape following the procedure described for hexagonal polycrystals in [3,4]. The line broadening of the rocking curves I (ω) is related to the local lattice desorientations and allows, therefore, an estimation of the excess density ρ_+ of edge dislocations introduced by the plastic bending of the crystals.

From the radial intensity distributions of the reflections on single crystals of different bending deformation states [2] total dislocation densities were estimated of $\rho \approx 10^9$ cm^{-2}. Rocking curves give excess dislocations densities in the range of $\rho_+ \approx 10^6$ cm^{-2}. This values are in good agreement with metallographic data found by the etch pit method and the value of geometrically necessary dislocations for plastically bending.

The scanning of the surroundings of the reciprocal lattice points in two directions gives therefore comprehensive informations about the produced substructure of the deformed crystal.

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X-RAY DIFFUSE SCATTERING IN HETEROSTRUCTURE: WAVEGUIDE MODES AND INTERFERENCE PHENOMENA

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The angular spectra of diffuse scattering by the rough surface contain the valuable information on the statistical characteristics of roughness. The different approaches have been developed to describe the diffuse scattering by multilayer nanostructures. Here we discuss the theory of the diffuse scattering by the arbitrary (periodic or irregular) multilayer structure with the rough interfaces. To calculate the amplitudes of diffusely scattered waves we have solved the boundary problem for single layer with two rough surfaces and then we have used the recurrence procedure for determination of the wave amplitudes. As a result the formulae for the intensity of the scattered radiation is obtained in the analytic form for an arbitrary number of layers. The general expression for the intensity of scattered radiation is applied to study the specific features of the diffuse scattering by the three- layer structure consisting of two *Fe* layers which are separated by layer of *C*. Such structure is used as a waveguide for the x-rays with the wavelength of a few angstroms.

The angular spectrum of the diffuse scattering depends significantly on the interlayer correlation function K_{nm} . We discuss the specific features of the angular spectra of scattering in the following cases: (a) the complete correlation (the correlation function does not depend on the index of layer, $K_{nm} = K$); (b) completely uncorrelated case, $K_{nm} = K_n \delta_{nm}$; (c) correlation of the first two interfaces, $K_{n3} = 0$ and $K_{nm} = K$ for $n, m = 1,2$. It is shown that the angular spectra of diffuse scattering depends significantly on the field distribution inside the structure and it is determined primarily by the integral field at the interfaces. For the small angles of incidence the integral field is a sum of the transmitted and reflected waves. As a result the angular spectrum of scattering depends on the angle of incidence, when $\theta_c^{(C)} < \theta_0 < \theta_c^{(Fe)}$, where $\theta_c^{(C,Fe)}$ are the critical angles of total external reflection for the *C* and *Fe* layers. We have shown that the waveguide modes are excited effectively by the diffuse scattering.

We have measured experimentally the angular spectra of specular reflection and diffuse scattering for the different three- layer structures.

ANGULAR SCANNING X-RAY TOPOGRAPHY INVESTIGATIONS OF HTS CRYSTALS IN ANOMALOUS TRANSMISSION REGIME

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Dynamical X-ray diffraction effects are known to exist only in high quality crystals. For example, the anomalous transmission effect, the Pendellosung effect, moire' interference, focusing of diffraction beams, ets. are realized only in semiconductors like silicon or germanium crystals and only a few others. The methods which are used for oservation of these effects need of the precize goniometers and collimated X-ray beams with a very small divergence.

It is known that new high temperature superconductor samples such as $ReBa_2Cu_2O_x$ (Re = Y, Ho, Cd ...) are offen bended and hence the use of ordinary methods to observations of dinamical diffraction in HTS crystals almost impossible. In our investigation of HTS crystals we have used a wide beam divergence method which was united with angular scanning method [1)] . In such combination the effect of the anomalous transmission of X-ray (the Borrmann effect) was observed in $Nd_2Cu_3O_4$, $(LaSr)_2CuO_4$, $EuBa_2Cu_3O_{7-x}$ and $YBa_2Cu_3O_{7-x}$ single crystals. With the help of it we have shown that the $EuBa_2Cu_3O_{7-x}$ crystals grown in an alundum container are noncentrosymmetrical. They have polarity axes along {100} and are devided into antiphase domains. We also obtained the result of the existence of the X-ray anomalous transmission effect in strongly distorted crystals. It shows that the theory of the Borrmann effect demands future development.

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PENDELLÖSUNG FRINGES OF SILICON AT LOW TEMPERATURES

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In connection with anomalous behaviour of the lattice spacing of the (444) plane of silicon that was found in recent X-ray diffraction experiments, we tried to examine the crystal structure at low temperatures. Since the anomaly is concerned with the state of a perfect crystal, and since we need high precision, the measurement of pendellösung fringes of X-ray diffraction has been used. By this method we can characterize perfect crystals, and determine precise absolute values of the crystal structure factor $|F|$ on the basis of the analysis of the period of intensity oscillation. Specimens, prepared from 200 μm thick (110) wafers of non-doped dislocation-free FZ silicon, were in a form of a rectangular plate 10 $\times$ 15 mm^2 in size; the longest edge was chosen to be parallel to the scattering vector. Measurements were made for the 111 diffraction with AgKα radiation (λ = 0.0561 nm) in a temperature range 17-300 K, and repeated over several thermal cycles. The effective thickness of the specimen was varied by turning the specimen about the scattering vector. For this, the specimen placed in a cryostat was mounted on a four-circle goniometer. The most serious technical problem was to keep the specimen, throughout the experiment, free from the effect of stress that deforms the pattern of pendellösung fringes. There were two main sources of such stresses: one is the difference in thermal expansion between the specimen and the holder on which the specimen was glued, and the other the mechanical vibration of the refrigerator type of cryostat. The pattern of pendellösung fringes was so sensitive to stresses that the problem was solved in a manner of trial and error. The results of the experiment show that the value of $|F|$ depends on the thermal history of the specimen at low temperatures; moreover, for cooling in the first thermal cycle, $|F|$ show a discontinuous change at 60 K to an extent of 3 %. These are qualitatively consistent with the results of the previous lattice spacing measurements. We therefore conclude that in the lattice of silicon crystals at the low temperature an atomic rearrangement occurs towards a more stable state with a different lattice spacing.

GAMMA-RAY DIFFRACTOMETRY INVESTIGATIONS OF DISLOCATION-CONTAINING QUARTZ SINGLE CRYSTALS.

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At present perfect method for the dislocation density determination does not exist. Actual techniques are destructive or require a long time. The aim of our works is development of the express non-destructive method allowing to determine the dislocation density with high accuracy. Another purpose is experimental testing of different modern theories of diffraction in real single crystals.

Gamma-ray diffractometry method has been applied for investigations of the quartz single crystals, containing dislocations with wide range of their density. Valuable density can be determined by broadening of the reflecting curves (the kinematical case of diffraction). By decrease of the dislocations density, this broadening is also decreased and becomes less than the angular resolution. In this case integral reflectivity is used. Strictly mathematically the problem of the dynamical scattering in dislocation-containing single crystals is not solved yet. A number of semi-empirical models have been suggested [1,2], but they have strong limitations and require certain preliminary supposition concerning defect structure.

The method based on the statistical dynamical theory of diffraction seems to be more fruitful. Primary theory proposed by Kato [3] can lead to inaccurate results in certain cases. Modifications of this theory [4-6] so far have been applied to rather perfect dislocationless silicon single crystals. In present work they are propagated to dislocation-containing, more imperfect quartz crystals, which permits to carry out experimental testing of the different versions of the statistical dynamical theory of diffraction.

Data obtained by means of these techniques will be compared with those obtained by other methods (X-ray topography, etching channel counting).

Developed technique allows to determine very low dislocation density with high accuracy (a few dislocations / cm^2).

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